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Development of a Mobile Treatment System for Handling Spilled Hazardous Materials

Envirex Inc.

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16. ABSTRACT

This report documents the results of a laboratory test program undertaken to define the treatment processes for the development of a modular transportable treatment unit for an on-site handling of spilled hazardous materials in aqueous solutions. The hazardous materials evaluated during this study were selected based on the priority ranking system developed by EPA. Nine materials evaluated for treatment by chemical reaction, clarification and activated carbon adsorption were: acetone cyanohydrin, acrylonitrile, ammonia, chlorinated hydrocarbons, chlorine, methanol, phenol, tetraethyllead (TEL) and tetramethyllead (TML). Several additional materials listed in the report were evaluated for treatment feasibility by reverse osmosis.

The results of the laboratory tests indicated that the unit treatment processes of chemical reaction, flocculation, sedimentation, granular media filtration and activated carbon adsorption would form the most suitable and versatile system for an on-site removal and treatment of hazardous materials. A 12.6 l/sec (200 gpm) mobile treatment system consisting of the above-mentioned processes was built based on the design data outlined in this report. This treatment vehicle is now ready and available for response to an actual or test spill and should be used at the earliest possible application opportunity.

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DEVELOPMENT OF A MOBILE TREATMENT SYSTEM FOR HANDLING SPILLED HAZARDOUS MATERIALS

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1. Environmental Health Effects Research
2. Environmental Protection Technology
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Copies of this report are available from the National Technical Information Service, Springfield, Virginia 22161.

FOREWORD

When energy and material resources are extracted, processed, and used, these operations usually pollute our environment. The resultant air, land, solid waste and other pollutants may adversely impact our aesthetic and physical well-being. Protection of our environment requires that we recognize and understand the complex environmental impacts of these operations and that corrective approaches be applied.

The Industrial Environmental Research Laboratory - Cincinnati assesses the environmental, social and economic impacts of industrial and energy-related activities and identifies, evaluates, develops and demonstrates alternatives for the protection of the environment.

This report is a product of the above efforts. It documents the laboratory and engineering studies conducted to determine the design of a Mobile Hazardous Material Spills Treatment Trailer. The report also contains a description of the "Trailer" and its main components (pumps, portable reaction and storage tanks, mixed media filters, activated carbon columns, etc.) and summarizes its spill response capabilities. The trailer, the fabrication of which is complete, is essentially a mobile self-contained filtration/carbon adsorption water treatment system which, for the first time, provides the capability to rapidly treat and remove spilled water soluble organics.

This report should be of interest to federal, state and local government personnel as well as to individuals from the chemical process and transportation industries who are faced with the problem of how to treat spills of hazardous chemicals in water.

David G. Stephan
Director
Industrial Environmental Research Laboratory
Cincinnati

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The support of the project by the U.S. Environmental Protection Agency, and the willing assistance and helpful advice of Project Officers Mr. Paul Minor and Dr. Joseph LaFornara in the conduct of the project is acknowledged with sincere thanks.

SECTION I

CONCLUSIONS

Based upon the laboratory data developed during this study for treatment of selected hazardous materials, it was concluded that:

1. A mobile waste treatment system consisting of chemical reaction, flocculation, sedimentation, granular media filtration and activated carbon adsorption would provide the most suitable and versatile system for on-site removal and treatment of hazardous materials.

2. Reverse Osmosis can be utilized on a selective basis for the treatment of water soluble inorganics. RO was found unsuitable for many hazardous materials because of their low water solubility and effect on membranes.

3. For the various hazardous materials evaluated in this study, it was found that:

    a. Activated carbon can be effectively used for the treatment of acetone cyanohydrin, acrylonitrile, chlorine and phenol.

    b. Chlorination can be used effectively for the oxidation of ammonia and for about 50% removal of acetone cyanohydrin and acrylonitrile.

    c. Most pesticides and herbicides (chlorinated hydrocarbons) can be removed by coagulation, filtration and carbon adsorption.

    d. Physical-chemical methods were not found to be suitable for the treatment of methanol. However, biological treatment may be applicable in some spill situations.

    e. TEL and TML can be effectively oxidized with potassium permanganate and also can be adsorbed partially via activated carbon after chemical flocculation, settling and filtration.

4. A 12.6 l/sec (200 gpm) mobile treatment system consisting of the treatment processes outlined in item 1 was constructed based on the design data outlined in this report.

SECTION II

RECOMMENDATIONS

It is recommended that:

1. The treatment vehicle built under this contract respond to a test spill to evaluate the effectiveness and capability of this treatment system.

2. Standard operating procedures and guidelines be developed for various hazardous materials, so that the hazardous spills vehicle can be deployed in an optimum manner in minimum time.

3. Trained personnel be employed to operate the vehicle and develop the standard operating procedures.

4. Multiple units similar to the 12.6 l/sec (200 gpm) mobile treatment system developed under this contract be built and placed at strategic locations around the United States for the on-site handling and treatment of spilled hazardous materials.

SECTION III

INTRODUCTION

"Hazardous substances" as a descriptive term covers a wide variety of materials, primarily because the word "hazardous" (like most qualitative adjectives) is essentially relative. Although many substances are obviously hazardous to aquatic and marine life due to direct inherent toxicity, other substances are equally detrimental to water ecosystems by virtue of indirect toxicity resulting from disturbance of the ecological balance such as the depletion of the dissolved oxygen supply required to support various life forms. Still others, although originally present at less than toxic or inhibitory levels, may become concentrated to these levels by residual accumulation through a succession of food chain transfers. Furthermore, even a substance as innocuous as molasses can become "hazardous", if a large quantity is released into a delicately balanced water ecosystem like a trout stream.

Handling and transport of these hazardous materials is necessary because of the wide spread usage of such materials in an industrial society. Although the essential feature of any overall program designed to minimize the risks involved in producing, handling, transporting, and using hazardous substances is prevention of spills, it should be recognized that spills will occur despite the most comphrehensive precautionary measures. When a spill does occur, the immediate primary concern is containment of the spilled material -- isolating the contaminating substance and preventing, in so far as possible, its encroachment on the surrounding environment. Ideally such containment will prevent the spilled material from reaching any water resources. However, such an ideal situation seldom exists in practice. Generally, containment is not possible, and a water body may be contaminated by the spilled hazardous materials which must therefore be purified. Thus, it is reasonable to expect that a development effort needs to be directed to minimize the effects of spilled hazardous materials that have reached a watercourse of some type (ditch, pond, stream, lake, river, etc.).

This concern for the accidental entrance of hazardous materials into a watercourse has initiated several programs by the Environmental Protection Agency for the control of this problem. The program described herein was undertaken to develop a transportable treatment system for on-site removal and treatment of spilled hazardous materials in aqueous solutions. This program has the following objectives:

1. Develop bench scale design data for the treatment of various hazardous materials.

2. Design and fabricate a mobile treatment system for spilled hazardous materials.

A listing of the hazardous materials evaluated during this study is shown in Table 1. This list was selected based on the priority ranking system for hazardous materials developed by EPA (1). As can be seen, the materials have been divided into two categories but overlap in several cases. Initially, only the nine materials listed in the first section of Table 1 were included for evaluation in the original contract 68-01-0099. However, later the above contract was amended to include a laboratory scale evaluation of the applicability of reverse osmosis for the pre-concentration of materials listed in the second section of Table 1. The intent of the laboratory evaluations was to screen the listed hazardous materials based on a brief review of available literature and to develop bench scale treatment feasibility data for those materials for which available information was lacking.

This report summarizes the laboratory, design and fabrication studies performed during the course of this project. Detailed engineering drawings and equipment specifications suitable for duplicating the mobile treatment system are in the possession of the Environmental Protection Agency as are a set of manuals with complete instructions for operating, repairing and maintaining the component devices. This comprehensive material can be made available to interested parties through EPA's Industrial Environmental Research Laboratory, Edison, New Jersey 08817.

Table 1. LIST OF HAZARDOUS MATERIALS

Materials considered in Selection of the Mobile Treatment System.

Acetone Cyanohydrin
Acrylonitrile
Ammonia
Chlorinated Hydrocarbon Pesticides
Chlorine
Methanol (Methyl Alcohol)
Phenol
Tetra Ethyl Lead (TEL)
Tetra Methyle Lead (TML)

Materials considered for reverse osmosis treatment feasibility.

Acetone
Acetone Cyanohydrin
Acrylonitrile
Alum (Aluminum Sulfate)
Ammonium Salts
Benzene
Chlorine
Chlorinated Hydrocarbon Pesticides (DDT, 2-4-D, 2-4-5-T)
Chlorosulfonic Acid
Copper Sulfate
Formaldehyde
Lead as in TEL and TML
Methanol
Mercuric Chloride
Phenol
Phosphorus Penta-Sulfide
Styrene

SECTION IV

TEST METHODS AND PROCEDURES

The unit treatment processes investigated for the hazardous materials listed earlier were chemical treatment, clarification via settling and/or filtration, activated carbon adsorption and reverse osmosis. These processes were selected based on the results of an earlier EPA study (2) which indicated that many of the hazardous materials could be precipitated by chemical treatment or oxidized to a more innocuous state by chemical treatment followed by sedimentation and/or filtration to remove the precipitated and other particulate matter such as soil or debris. In addition, activated carbon was found to be one of the most versatile and affirmative processes for the treatment of a wide variety of water soluble materials.

Reverse osmosis treatment feasibility evaluations were based on the promise of utilizing RO in conjunction with activated carbon. It was thought that this combination could provide tremendous versatility of treatment for both inorganic and organic hazardous materials. Reverse osmosis was promising as a pre-concentration step prior to activated carbon treatment for various organic materials because of its compact size, high volume and low weight in a mobile treatment system. Reverse osmosis treatment could also be utilized for the treatment of various inorganics, such as toxic heavy metals, that otherwise could not be treated via activated carbon.

A brief description of the various bench scale treatment test procedures employed is presented in the following sections. A description of the laboratory procedures and the analytical apparatus utilized in this study is included in Appendix A. Bench scale treatment tests were conducted only in those areas where available information from literature was lacking.

CHEMICAL TREATMENT AND CLARIFICATION

The bench scale chemical treatment tests were generally conducted in 100 to 1,000 ml beakers and graduated cylinders. Chemicals were added in measured quantities according to preselected dosages and suitable reaction times were provided to evaluate optimum chemical dosages. Sedimentation time generally ranged between 30 to 60 minutes. The pollutant concentrations were analyzed after clarification to evaluate the degree of removal achieved during various tests. The hazardous materials for which the

chemical treatment tests were conducted were: acetone cyanohydrin, acrylonitrile, ammonia, chlorinated hydrocarbons, chlorine, phenol, TEL and TML.

ACTIVATED CARBON ADSORPTION

Both isotherm and dynamic column type carbon adsorption tests were conducted to supplement existing data for the selected hazardous materials listed in Table 1. A detailed description of the bench scale procedures for the two types of carbon tests is included in Appendix B. The materials for which the carbon adsorption was evaluated were: acetone cyanohydrin, acrylonitrile, chlorinated hydrocarbons, chlorine, methanol, phenol, TEL and TML. All carbon tests were conducted with filtrasorb 400 manufactured by Calgon Co., Pittsburgh, Pennsylvania.

REVERSE OSMOSIS TREATMENT

Reverse osmosis treatment feasibility tests were conducted with a hollow fine fiber membrane made of a polyamide nylon and designated as B-9 by the manufacturer, Du Pont Co. A half size B-9 permeator (membrane and the pressure vessel) with a nominal capacity of 0.09 l/sec (2100 gpd) was utilized for these studies.

The test solutions were made in tap water in desired concentrations. Membrane rejection capabilities were evaluated by analyzing the raw product and brine streams for pertinent constituents. Flow rates, temperatures and pressures were recorded during each test. The tests were generally of short duration and lasted between 1 to 4 hours. A detailed description of the test setup and procedures is shown in Appendix B. All the materials listed in the second section of Table 1 were screened for RO feasibility tests based on available literature; however, actual RO tests were conducted for acetone, acetone cyanohydrin, acrylonitrile, ammonium nitrate, cooper sulfate, formaldehyde, methanol and mercuric chloride.

SECTION V

TEST RESULTS AND EVALUATIONS

The test results and data have been divided into two separate sections as listed earlier in Table 1. The nine materials considered in the selection of the mobile treatment system are discussed first for chemical treatment, clarification and carbon adsorption treatment. A second section presents the results of the RO feasibility tests.

ACETONE CYANOHYDRIN

It was indicated in the literature (2) that acetone cyanohydrin may be detoxified at high pH levels by the precipitation of cyanides via chlorination or carbon adsorption treatment. Therefore, chlorination and carbon adsorption tests were conducted for this material.

Chlorination Tests

Solutions containing approximately 900 mg/l acetone cyanohydrin were treated with sodium hypochlorite at ratios of 2.9 and 1.1 parts of chlorine per part of acetone cyanohydrin at pH values of 9, 10, 11, and 12 (Table 2) for 30 minutes. Chlorine concentrations were measured by iodometric titration and acetone cyanohydrin was measured by gas chromatography. Sodium hydroxide was used to adjust the pH at the start of the tests. From these tests it was found that chlorine dosages of at least 3 parts chlorine per part of acetone cyanohydrin must be used at a pH of at least 12 to achieve any significant reduction (about 50%) in acetone cyanohydrin concentration. This treatment therefore, was not considered to be a feasible method of treatment for spills of this material. Additional tests were conducted with anthium-dioxide. Anthium dioxide is a stable solution containing 50,000 mg/l chlorine dioxide. Chlorine dioxide is reported to be a more powerful oxidizing agent than chlorine and results in fewer chlorinated byproducts. The anthium dioxide sample tested had an oxidation capacity equivalent to 145,000 mg/l chlorine as determined by iodometric titration. The dosage of anthium-dioxide used in these tests is expressed here in terms of equivalent chlorine concentration to facilitate comparison with the previous sodium hypochlorite test work. The results of tests performed on solutions containing about 8,000 mg/l acetone cyanohydrin are listed in Table 3. These tests were performed using ratios of

Table 2. OXIDATION OF ACETONE CYANOHYDRIN WITH NaOCl - 30 MINUTE CONTACT TIME

Test No.	A	B	C	D	E	F	G	H
Acetone cyanohydrin conc., mg/l								
at start	870	870	864	850	903	898	895	883
after treatment	862	815	489	420	850	799	828	768
removed	8	5	375	430	63	99	67	115
NaOCl conc., mg/l as Cl_2								
amount added	2500	2500	2480	2440	1035	1030	1030	1010
after treatment	645	576	35	16	18	9	13	9
used	1855	1924	2445	2424	1017	1021	1017	1001
pH at start	9.3	10.0	11.0	12.0	9.0	10.0	11.0	12.0
pH at end	9.3	10.1	11.6	12.2	8.4	10.9	10.8	11.9
Wt/wt ratios:								
Cl_2 added/acetone cyanohydrin present	2.88	2.88	2.88	2.88	1.15	1.15	1.15	1.15
Cl_2 used/acetone cyanohydrin removed	232	35	6.50	5.60	16	10	15	9

Table 3. OXIDATION OF ACETONE CYANOHYDRIN WITH CHLORINE DIOXIDE

Test No.	1	2	3	4
Acetone cyanohydrin conc., mg/l				
at start	8660	8070	7600	8190
after 0.5 hours	8120	7440	5240	7460
after 1.5 hours	7000	5400	4980	4160
Acetone cyanohydrin removed, mg/l				
after 0.5 hours	540	630	2360	730
after 1.5 hours	1660	2670	2620	4030
Anthium dioxide conc., mg/l as Cl_2				
added to sample	5400	10000	14200	10200
after 0.5 hours	4752	8153	4927	8720
used in 0.5 hours	648	1847	9273	1480
pH at start	5.00	5.00	5.00	6.00
pH at end	5.00	5.10	4.40	5.90
Wt/wt ratios:				
Cl_2 added/acetone cyanohydrin	0.62	1.24	1.87	1.24
Cl_2 used/acetone cyanohydrin (1/2 hr)	1.20	2.90	3.90	2.00

chlorine and acetone cyanohydrin of 0.6:1 to 1.9:1 at pH levels of 5 and 6. The chlorine dioxide is released by reducing the pH of the solution to less than 7; the lower the pH, the faster the reaction. After 30 minutes, the amount of chlorine dioxide remaining was measured by iodometric titration. Acetone cyanohydrin concentration was measured by gas chromatography at thirty minutes and 90 minutes. It appears that relatively long contact times are needed (more than 1/2 hour) for treatment at the pH levels tested. Lower pH levels are undesirable because of the hazards of cyanide formation. For these reasons and since only a maximum of 50% removal was achieved, anthium dioxide was not given further consideration as an acetone cyanohydrin spill counter measure.

Activated Carbon Treatment

Isotherm tests were performed on two solutions containing 888 mg/l and 9120 mg/l acetone cyanohydrin in tap water. .Lime was added to each solution to raise the pH to 11 to prevent hydrogen cyanide gas formation. The solutions were filtered prior to running the isotherm tests. The results of the isotherm tests are plotted in Figure 1. The estimated adsorption capacity for the two solutions were 0.27 mg/mg carbon for the 888 mg/l solution.

Two carbon column tests were also made on acetone cyanohydrin at feed concentrations of 9,225 mg/l and 95 mg/l. The results of the first test, at a hydraulic loading rate of 3.27 l/sec/m^2 (4.8 gpm/ft^2) and a concentration of 9200 mg/l is shown in Figure 2. Nearly complete removal of acetone cyanohydrin was achieved. The adsorption capacity was found to be 0.203 mg/mg carbon compared to the isotherm value of 0.27 mg/mg carbon.

The second carbon column run with acetone cyanohydrin was made at a feed concentration of 95 mg/l and a hydraulic loading of 2.7 l/sec/m^2 (4.1 gpm/ft^2). The results of this test are presented in Figure 3. Again, complete removal of acetone cyanohydrin was achieved. The adsorption capacity was found to be 0.016 mg/mg carbon or 85% of the carbon isotherm value (0.019 mg/mg carbon). During the second run the presence of cyanide in the effluent was also investigaged. Cyanide was found to be present in measurable quantities as shown in Figure 3. This presents a problem, as cyanide is toxic, and is evidently released by the adsorption reaction. However, cyanide can be precipitated by the ferric ion (2).

As evidenced by the carbon column breakthrough curves, activated carbon effectively reduces the discharge of acetone cyanohydrin and can be used for such spills. Minimum contact time was found to be approximately 50 minutes.

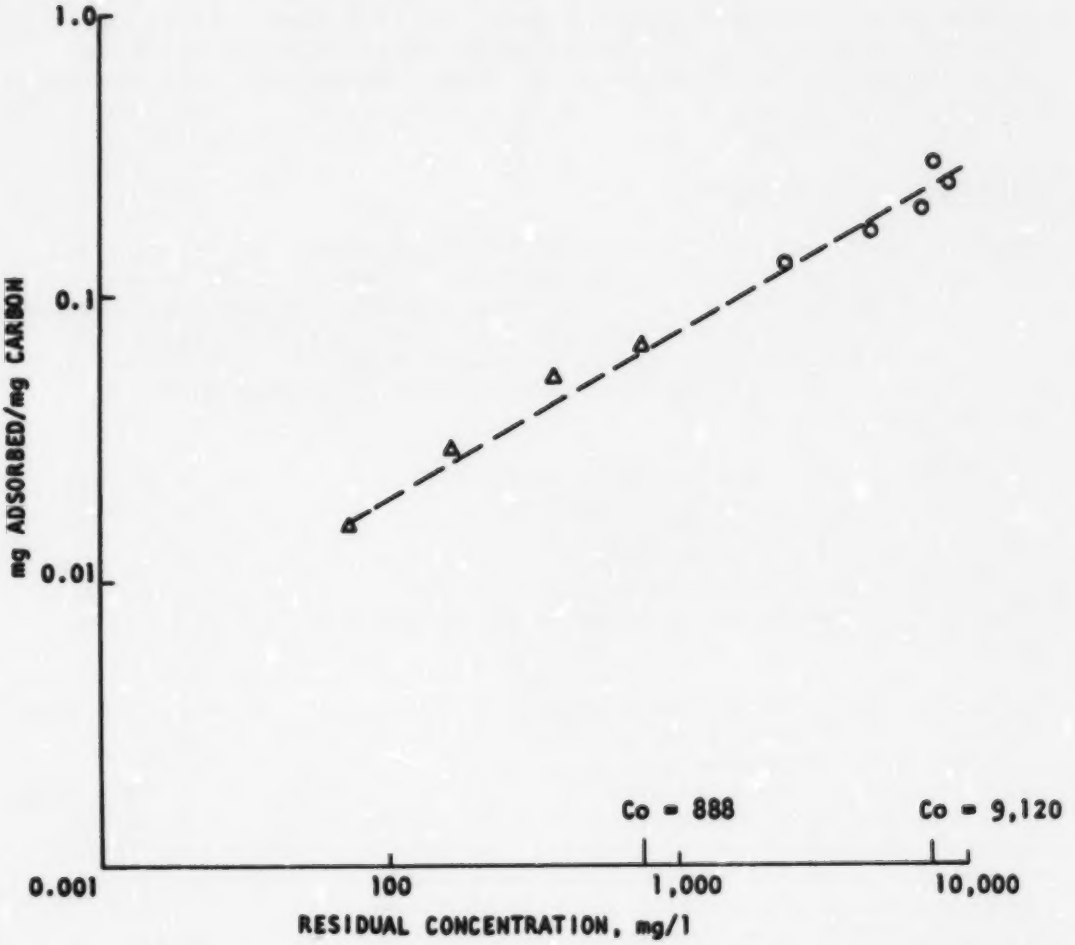


Figure 1. Carbon isotherm results for acetone cyanohydrin

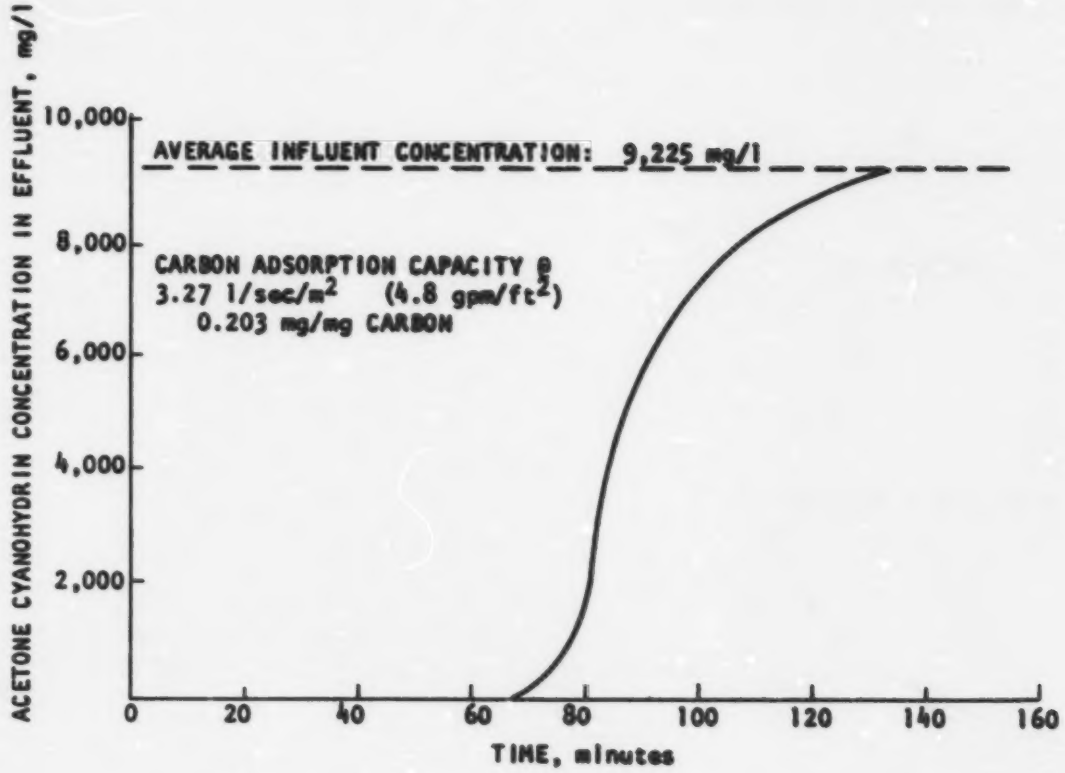


Figure 2. Carbon column test for acetone cyanohydrin (high concentration)

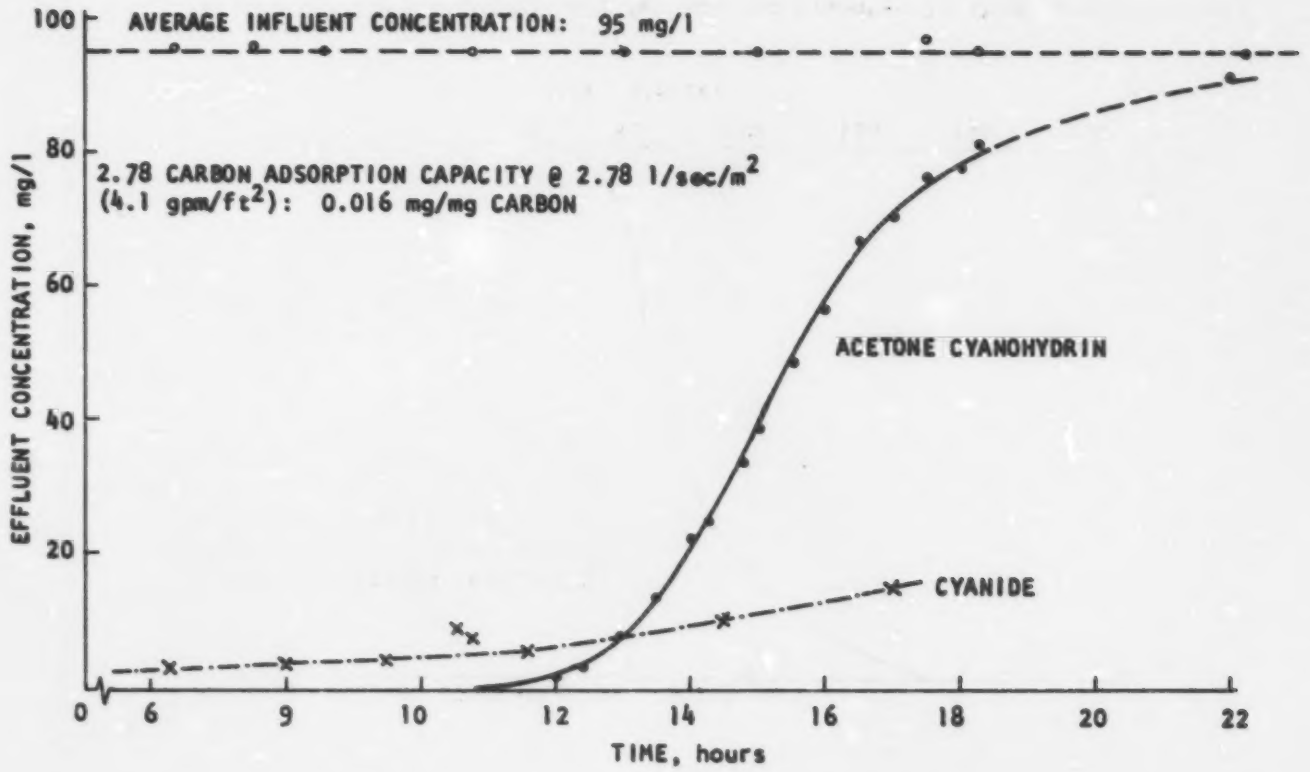


Figure 3. Carbon column test for acetone cyanohydrin (low concentration)

ACRYLONITRILE

Literature (2) indicated that this compound must also be handled at elevated pH values of above 9.0 to prevent the formation of hydrogen cyanide gas. Chlorination and activated carbon adsorption were again indicated to be viable methods of treatment for this compound. Since this material was considered to be quite volatile, tests were also conducted to study the volatalization of this solution prior to chemical treatment tests.

Volatility Tests

Two solutions containing an initial concentration of 7600 mg/l acrylonitrile were allowed to stand in open beakers at room temperature. One solution had a pH of 7.8 and the other had a pH of 11.7. The ratio of solution surface to volume was approximately 0.08 m^2/l (3.3 $ft^2/gal.$). Samples were removed periodically for analysis of acrylonitrile. The rate loss of acrylonitrile was the same for each solution; the loss followed a first-order reaction with a K constant of 0.015 min^{-1}. The "half life" (elapsed time required for loss of half the acrylonitrile) was 47 minutes at ambient temperature. A check for cyanide ion showed that the acrylonitrile loss was not due to cyanide formation in either solution. Although conditions of temperature, wind and ratio of surface area to volume of contaminated water may vary widely for different spill situations, it is quite likely that appreciable quantities of acrylonitrile will be vaporized before carbon adsorption equipment is ready to treat the contaminated water.

Because of the above observation, all further chemical tests for acrylonitrile were performed in stoppered erlenmeyer flasks equipped with a septum such that samples for analysis could be withdrawn with a syringe to insure reproducibility.

Chlorination Tests

Three tests were performed to determine the effect of chlorination on gas chromatograph acrylonitrile solutions. Solutions containing 5900 mg/l, 4300 mg/l and 353 mg/l acrylonitrile were used. Sodium hypochlorite was added to these solutions at ratios of 0.3, 1.4 and 16.4 mg per mg acrylonitrile. The remaining acrylonitrile and chlorine concentrations were measured after one hour of contact. The results of these tests are listed in Table 4. The amount of chlorine required varied from 2.5 to 4.9 mg chlorine per mg acrylonitrile destroyed. It should be noted, however, that as the concentration of acrylonitrile decreased, other compounds were formed as indicated by the appearance of two additional peaks on the chromatogram. Although it was not possible to establish

the identity or quantity of the two byproduct compounds, an approximation of the relative amount of material obtained from the area of the two peaks (75 mm^2) relative to the acrylonitrile peak (188 mm^2) demonstrate that a large portion of the acrylonitrile was converted to other organic materials which will require further treatment. Because of the need for large chlorine dosage and the production of byproducts, further work with sodium hypochlorite was not done.

A few tests were performed to investigate the effect of ferric ion on acrylonitrile. No appreciable reduction in acrylonitrile was observed. Iron salts may have an application only if the acrylonitrile has hydrolyzed (in nature) to form cyanide. It is not recommended that hydrolysis be induced if it has not occurred via natural processes.

Table 4. CHLORINATION OF ACRYLONITRILE

	Test 1	Test 2	Test 3
Initial acrylonitrile conc., mg/l	5900	4330	353
pH	10.8	10.7	8.9
Acrylonitrile conc. at 1 hr, mg/l	5600	3120	165
Acrylonitrile removed, mg/l	300	1210	188
Chlorine added, mg/l	1640	6015	5800
Chlorine at 1 hr, mg/l	560	3170	4885
Chlorine used, mg/l	1080	2845	915
Chlorine added: Acrylonitrile added	0.3:1	1.4:1	16.4:1
Chlorine used: acrylonitrile removed	3.6:1	2.4:1	4.9:1

Carbon Adsorption Tests

Carbon isotherm tests were performed on three solutions of acrylonitrile diluted with tap water with a minimum pH of 9. Isotherm tests were performed on solutions containing 73, 805 and 7600 mg/l acrylonitrile. The results of these tests are plotted in Figure 4. The estimated adsorption capacities from this figure are: 0.25 mg/mg carbon for the 7600 mg/l solution; 0.072 mg/mg carbon for the 805 mg/l solution and 0.019 mg/mg carbon for the 73 mg/l solution.

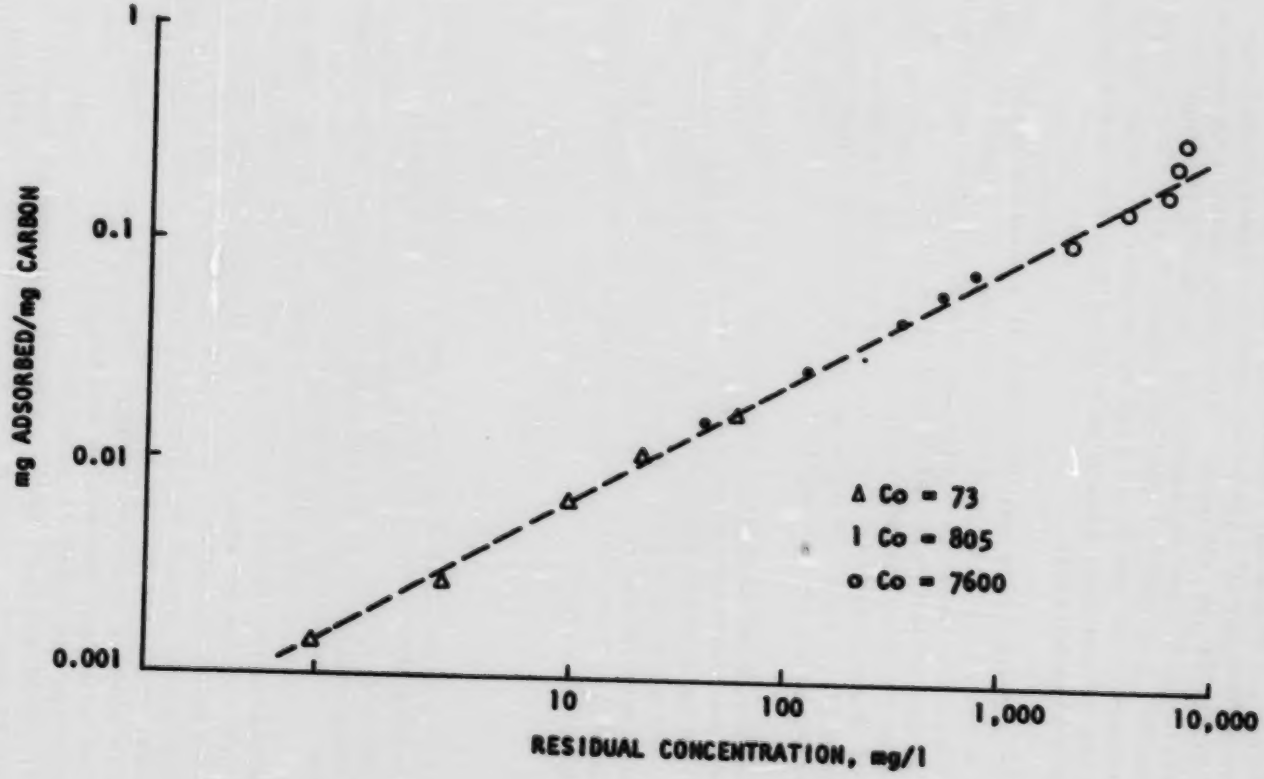


Figure 4. Carbon isotherm results for acrylonitrile

Carbon column tests with acrylonitrile at a concentration of approximately 600 mg/l were performed. Two hydraulic rates utilized were 1.8 and 3.4 l/sec/m^2 (2.7 and 5.0 gpm/ft^2). The results of these tests are presented in Figure 5. There was essentially no difference in the adsorption capacity as a function of hydraulic loading. Nearly complete removal of acrylonitrile was achieved. Adsorption capacities were 0.143 and 0.134 mg acrylonitrile per mg carbon for hydraulic loadings of 3.4 and 1.8 l/sec/m^2 (5.0 and 2.7 gpm/ft^2) respectively. The carbon isotherm for acrylonitrile predicted 0.22 mg acrylonitrile per mg of carbon. Hence the column test showed only about 63% adsorption capacity compared to the isotherm value.

A second test of acrylonitrile was conducted at a lower concentration, 70 mg/l and at a hydraulic loading of 2.9 l/sec/m^2 (4.3 gpm/ft^2). The results of this run are shown in Figure 6. The adsorption capacity, as determined by the column test was 0.0142 mg acrylonitrile per mg carbon as compared to the isotherm value of 0.019. It should be noted that the rate of breakthrough of acrylonitrile through the carbon column at the lower concentration of 70 mg/l was significantly slower than at the higher concentration of 6000 mg/l.

This testing demonstrated that activated carbon could be utilized effectively for the treatment of acrylonitrile solutions. Minimum contact time was found to be approximately 40-50 minutes. Because of the volatile nature of acrylonitrile, a significant amount will evaporate to the atmosphere and create an air pollution hazards. It is therefore especially imperative that rapid action be taken by personnel protected by adequate safety equipment.

AMMONIA

Dawson et al (2) indicated that the most applicable technique for the handling of ammonia spills is dilution. It is also indicated that stripping of ammonia at high pH values may be applicable in certain situations. Chlorination can also be used for the oxidation of ammonium ion to nitrogen. Information concerning the oxidation of dilute concentrations of ammonia with chlorine is available in the literature. Generally, weight/weight ratios of Cl_2:NH_3 of 4:1 to 10:1 are recommended for complete destruction of ammonia. Lower concentrations of chlorine results in chloramine formation. The reaction is reported to be fastest at pH 8.3. The actual amount of chlorine needed at a spill site would vary, of course, depending on the amount of other materials in the water that would also exert a chlorine demand. One test was performed on the chlorination of solutions containing approximately 850 mg/l of ammonia. Sodium hypochlorite was added to solutions of ammonium chloride at ratios of 1.16, 2.92 and 5.85 parts chlorine per part of ammonia at pH values of 8.5, 11 and 12. After a thirty minute reaction period, the amount of chlorine remaining was measured by iodometric titration.

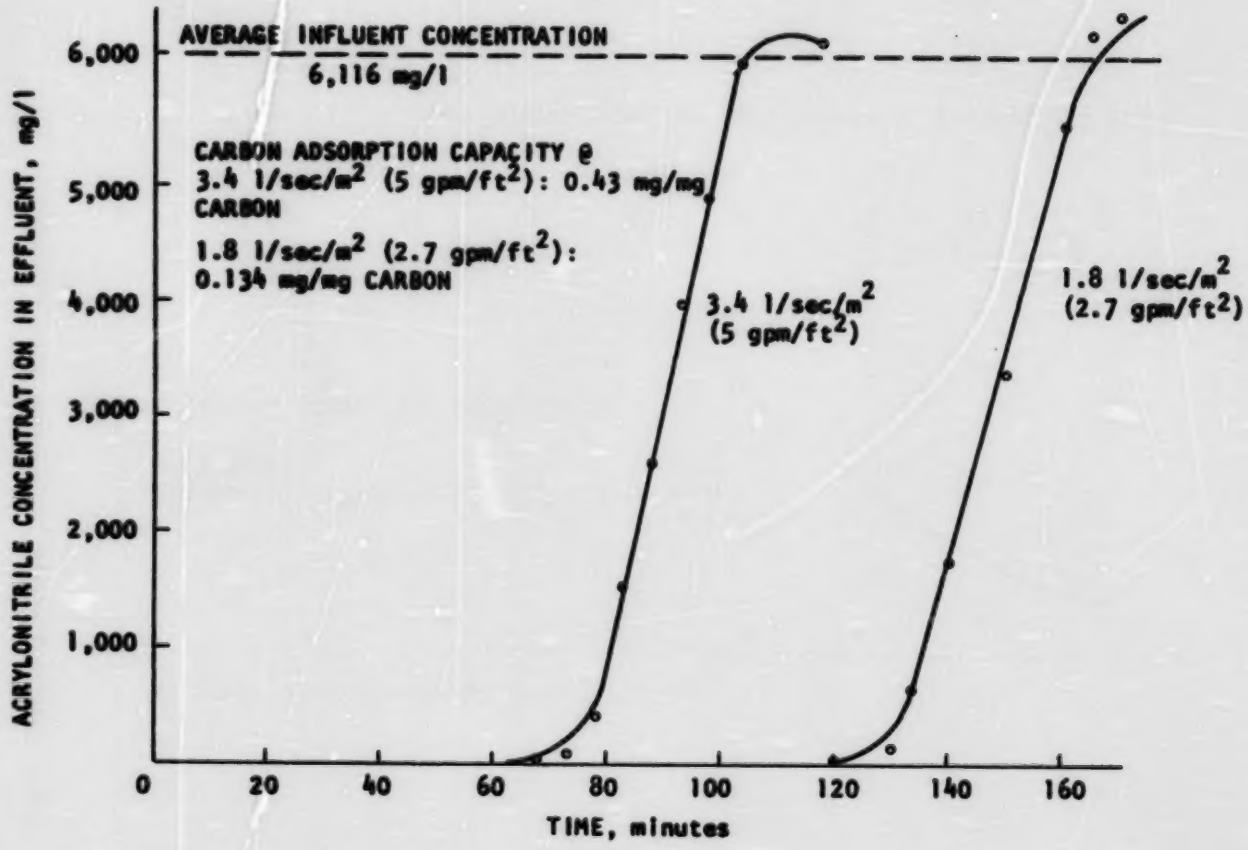


Figure 5. Carbon column tests for acrylonitrile (high concentration)

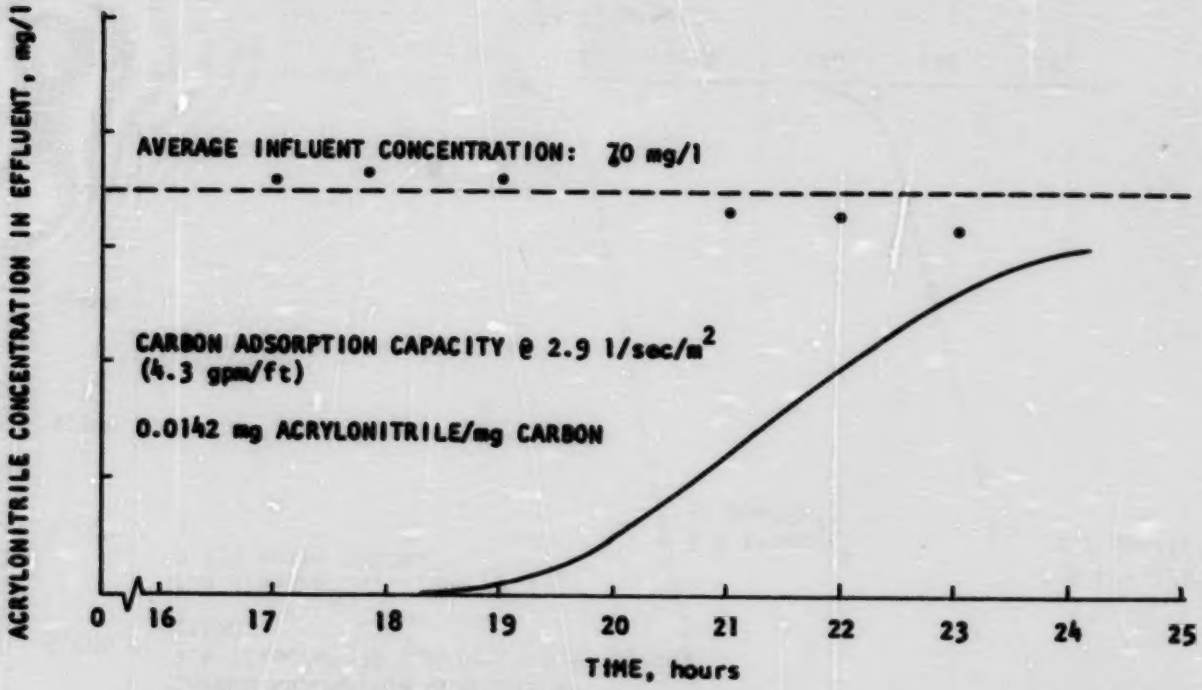


Figure 6. Carbon column tests for acrylonitrile (low concentration)

A portion of the sample was then dechlorinated and the remaining ammonia and the chloramines converted to ammonia by the dechlorination and measured by nesslerization.

The results are listed in Table 5. At pH 8.5, a Cl_2:NH_3 ratio of 5.85 was needed for a significant reduction in ammonia. There was no ammonia destruction at lower ratios at a pH level of 8.5. Some ammonia was however, destroyed at a ratio of 2.9:1 at higher pH values. Although some of the ammonia may have been lost by volatilization at the high pH values, the amount of chlorine used indicated some destruction. Recent literature (4) indicates that a 7:1 to 10:1 Cl_2NH_3 ratio is needed to effectively remove all the ammonia. Hence, ammonia removal by breakpoint chlorination is chemically feasible for relatively dilute concentrations of ammonia spills, but such treatment does not appear to be logistically possible owing to the large amounts of chlorine required and the secondary pollution it would cause.

CHLORINE

Activated carbon may be utilized for the dechlorination of chlorine contaminated solutions. One isotherm test has been performed on a sample of sodium hypochlorite containing 9290 mg/l chlorine. The results of this test are plotted in Figure 7. The estimated adsorptive capacity at 9290 mg/l was 1.7 mg chlorine/mg carbon.

Column tests were performed on a 9000 mg/l solution of chlorine to further investigate the feasibility of chlorine removal by activated carbon at a hydraulic loading rate of 3.4 l/sec/m^2 (5 gpm/ft^2). An adsorption capacity of 2.5 mg Cl_2/mg carbon was achieved, which was greater than predicted by the isotherm tests. The minimum contact time was found to be of the order of only a few minutes. It is indicated that chlorine removal by activated carbon is not an adsorption phenomenon but an oxidation-reduction reaction. Chlorine is reduced to chloride utilizing an activated carbon molecule which in turn gets oxidized to carbon dioxide. This production of CO_2 causes an extra buildup of pressure inside the carbon column and may cause short circuiting by gas pocket formation. The treated carbon column effluent was a characteristic dark brown color imparted by the extremely fine carbon particles produced due to the oxidation of activated carbon. The color of the treated effluent was much less intense, however, when the influent waste pH was controlled between 5 and 6. Dilution of chlorine spills prior to carbon treatment might also minimize this problem. It is therefore indicated that granular activated carbon treatment is generally feasible for mitigating chlorine spills.

Table 5. CHLORINATION OF AMMONIA WITH NaOCl - 30 MINUTE CONTACT TIME

Test No.	1	2	3	4	5
Ammonia conc., mg/l as NH_3					
at start	877	946	961	820	810
at end	875	875	290	426	398
removed	2	--	571	394	412
NaOCl conc., mg/l as Cl_2					
added	1020	2470	5030	2390	2360
at end	830	830	600	727	332
used	190	1640	4430	1663	2028
pH at start	8.50	8.50	8.50	11.00	12.00
pH at end	8.35	6.10	5.70	10.80	12.00
Wt/wt ratios:					
Cl_2 added/NH_3 present	1.16	2.92	5.85	2.92	2.92
Cl_2/NH_3 removed	--	--	--	--	--

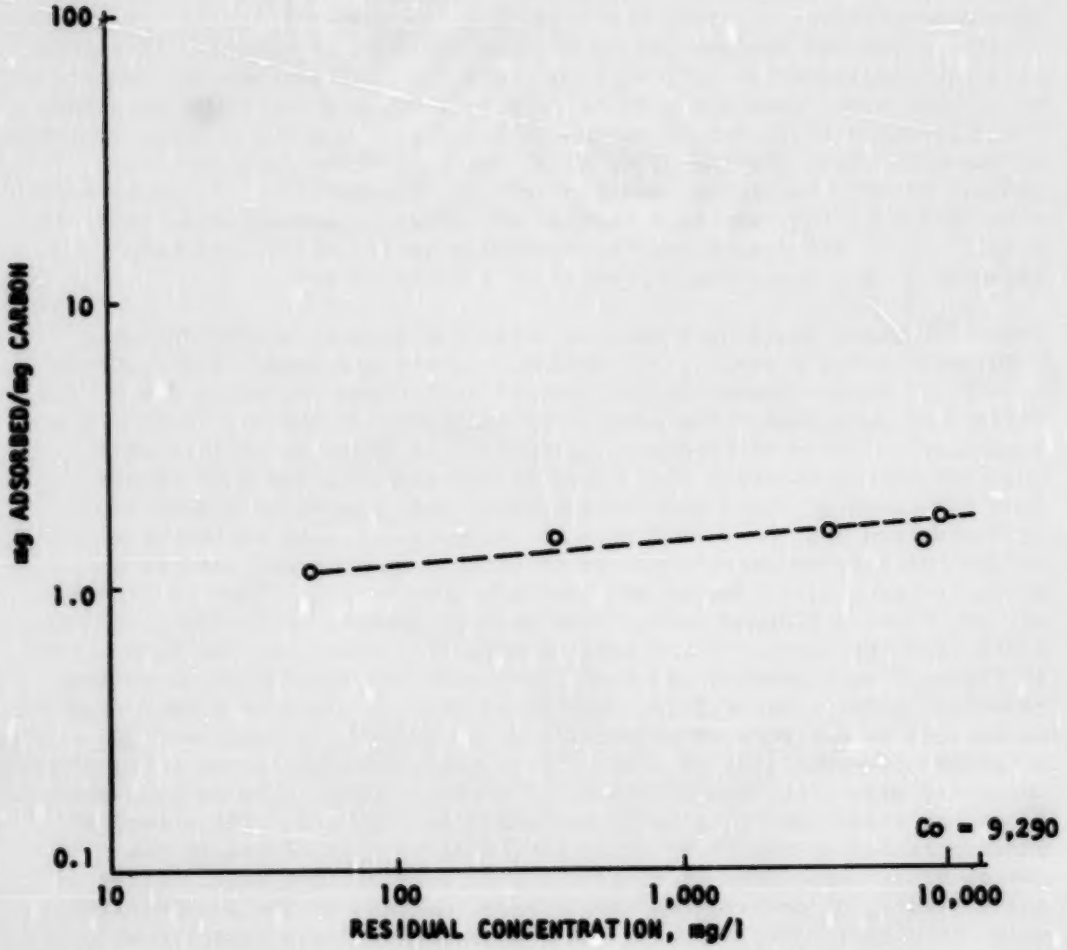


Figure 7. Carbon isotherm results for chlorine

CHLORINATED HYDROCARBONS

While activated carbon adsorption was anticipated to be among the most effective methods for the removal of most spilled chlorinated hydrocarbons, pesticides and herbicides, a brief review of the literature showed that a great deal of work has also been done on the adsorption of pesticides on various other materials. The data of King, Yeh, Warren and Randall (Table 6) show substantial removals of lindane from aqueous solutions when concentration of soils was high (8). In contrast, Lotse, Graetz, Chesters, Lee and Newland (Table 6) found much lower removal efficiency at a low concentration (372 mg/l) of soil (9). Efficiency was reported to be even lower when soils containing less organic materials was used. The adsorption of 2, 4-D on montmorillionite clay was found to be minimal by Schwartz (10). On the other hand, Huang and Liao have reported removal of DDT, heptachlor and dieldrin using clay (11). It appears from this data that the removal of suspended solids in contaminated water at a spill site will remove the chlorinated organics which have been adsorbed on the suspended particles of a suitable soil.

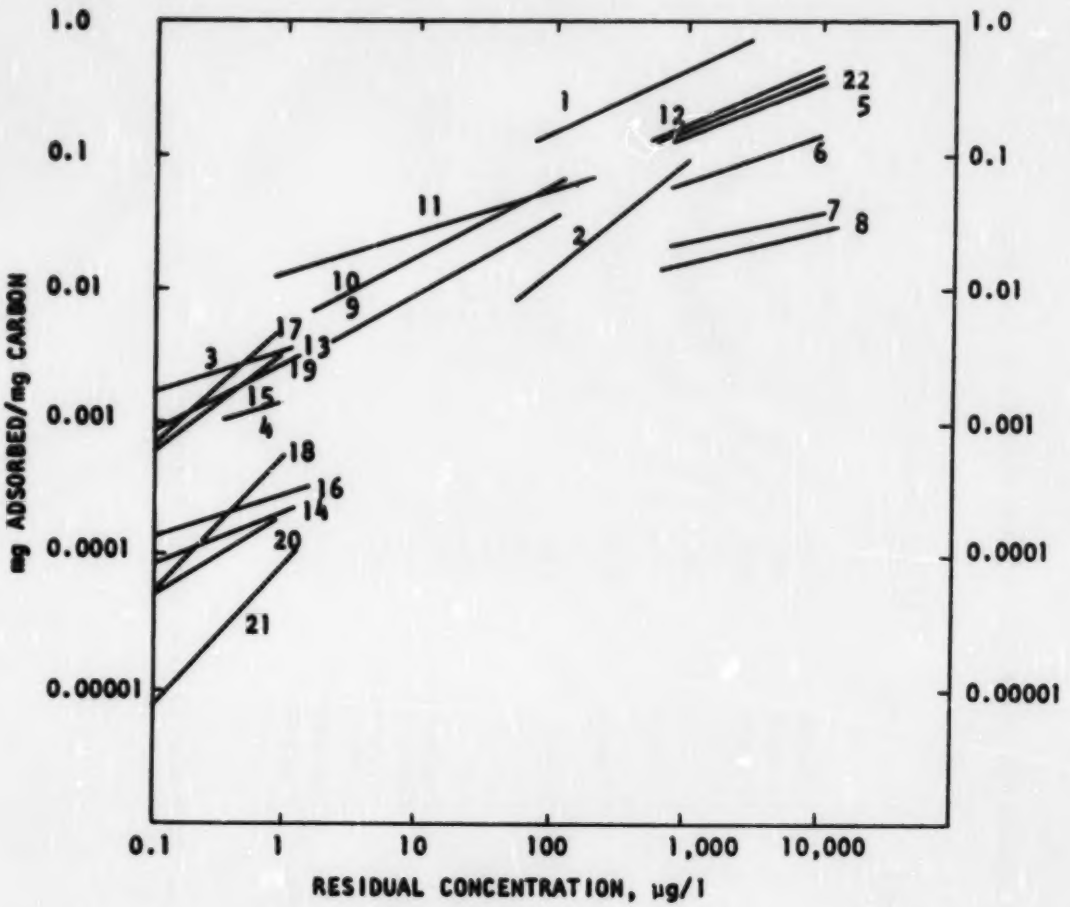
Carbon isotherm tests have been reported for several pesticides and herbicides using a variety of carbons. Morris and Weber (12) reported results of carbon adsorption on several herbicides including 2,4-D, 2,4,5-T, Silvex and parathion. The adsorption isotherms in the Morris and Weber papers are plotted as Langmuir isotherms. In order to be consistent with the carbon isotherm plots used in our studies, the data points from the Langmiur plots were recalculated and plotted as Freundlich isotherms and are shown in Figure 8. King, et al also performed a carbon isotherm on parathion. In this test, acetone was used as a bridge solvent (8). The carbon isotherm plot is also shown in Figure 8. Aly and Faust performed carbon isotherms on several herbicides including 2,4-D and the iso-octyl and butyl esters of 2,4-D. The isotherm plots in Figure 8 were constructed from the Freundlich adsorption parameters reported in their paper (13). Carbon adsorption tests were performed on 2,4,5-% butoxy-ethanol ester, dieldrin, endrin, lindane, and parathion by Robeck, et al (14). These tests were performed using distilled water and water from the Little Miami River in Ohio. The carbon isotherm lines for these herbicides are also shown in Figure 8. The effect of other organics in the river water on the adsorption of pesticides (as on other compounds) is clearly illustrated by this data. It is quite likely, of course, that the organic content of the contaminated water at a spill site may be quite high and that these organics will compete with the hazardous material for the activated carbon, and that the pesticides and herbicides will not be well adsorbed. All the isotherms mentioned are plotted in Figure 8 to help estimate the carbon treatment capacity of these materials.

Very little information was found in the literature concerning the adsorption of DDT. Although Huang and Liao reported on the adsorption of DDT on various clays, no reference was found concerning the adsorption on activated carbon (11). Indeed, information concerning the

Table 6. ABSORPTION OF PESTICIDES AND HERBICIDES ON SOILS

Clay or soil type	Clay or soil concentration	Pesticide used	Pesticide concentration, initial, µg/l	Pesticide concentration, final, µg/l	Contact time, hours	Reference[a]
Berks	3%	Lindane	3600	100	1	8
Berks	3%	Lindane	196	0.1	1	8
Cecil	7%	Lindane	620	100	1	8
Cecil	7%	Lindane	40	0.1	1	8
Florida	7%	Lindane	500	100	1	8
Florida	7%	Lindane	36	0.1	1	8
64% Organic	372 mg/l	Lindane	1860	1610	24	9
Montmorillonite	800 mg/l	2, 4-D	130	129	23	10
Illite	1000 mg/l	DDT	100	19	1	11
Kaolinite	1000 mg/l	DDT	100	24	1	11
Montmorillonite	1000 mg/l	DDT	100	13	1	11
Illite	1000 mg/l	Heptachlor	100	60	1	11
Kaolinite	1000 mg/l	Heptachlor	100	40	1	11
Montmorillonite	1000 mg/l	Heptachlor	100	40	1	11
Illite	1000 mg/l	Dieldrin	100	74	1	11
Kaolinite	1000 mg/l	Dieldrin	100	75	1	11
Montmorillonite	1000 mg/l	Dieldrin	100	75	1	11

a. Reference numbers refer to the numbered literature references in Section VII.



1. Parathion
2. Parathion
3. Parathion - D.W.
4. Parathion - river
5. 2,4-D
6. 2,4-D at pH 3
7. 2,4-D at pH 7
8. 2,4-D at pH 11
9. 2,4-D
10. 2,4-D buthyl ester
11. 2,4-D iso cotyl ester
12. 2,4,5-T
13. 2,4,5,-T butoxyethenol ester in distilled water
14. 2,4,5-T butoxyethanol ester in river water
15. Lindane - D.W.
16. Lindane - river
17. Endrin - D.W.
18. Endrin - river
19. Dieldrin - D.W.
20. Dieldrin - river
21. DDT
22. Silves

Figure 8. Carbon adsorption isotherms for various pesticides & herbicides

solubility of DDT is confusing. Hartung and Klingler (15) cite references (16)(17) which report the solubility of p,p'-DDT as being 37 µg/l and 1.2 µg/l respectively. Robeck, et al (14) filtered a suspension of a technical grade of DDT (containing o,p-DDT, p,p'-DDT and DDE) through a 0.05 µ filter and a 5 µ filter. The total concentration of the two DDT isomers and DDE passing the filters was 16 µg/l and 40 µg/l respectively. It was concluded that the apparent solubility was due to the size of pesticide particles remaining in suspension.

Because of the scarcity of data regarding DDT, a carbon isotherm test was performed in the laboratory. A one gram portion of p,p'-DDT was mixed with three liters of tap water and was allowed to sit for seven days. The DDT remaining in suspension was removed by filtering through an 0.45 µ membrane filter. Analysis of the filtrate for p,p'-DDT revealed less than 0.2 µg/l concentration. Because of this, a second mixture of DDT and water was prepared and mixed for five days. A 200 ml portion was filtered through a 0.45 µ membrane filter and another 200 ml portion was filtered through a Type A Gelman glass fiver filter (47 mm diameter). Again, no p,p'-DDT was found in the filtrate from the membrane filter (<0.2 µg/l). The filtrate from the glass fiber filter contained 61 µg/l p,p'-DDT. The remaining mixture, approximately 2.5 liters in volume, was filtered through a second glass fiber filter and was found to contain 26 µg/l p,p'-DDT. This filtrate was used for the carbon isotherm tests.

The carbon isotherm tests were performed in 250 ml erlenmeyer flasks containing 200 ml filtrate, a selected volume of carbon slurry (powdered Filtrasorb 400) and distilled water to bring the total volume of 210 ml. The two carbon slurries used contained 1 mg carbon/ml and 10 mg carbon/ml respectively. The isotherm flasks contained 0, 0.1 mg, 1 mg, 10 mg and 100 mg carbon. After one hour of shaking on a Burrell shaker, the contents of each flask were filtered through a clean glass fiber filter. The filtrates were analyzed for p,p'-DDT. The results of this isotherm test are plotted in Figure 9. A reduction in DDT concentration from 26 µg/l before the isotherm test, to 5.1 µg/l in the sample containing no carbon may have been due to removal of more DDT by the second filtration. However, the adsorption capacity demonstrated in Figure 9 is 0.00053 mg DDT/mg carbon. Evidently activated carbon does adsorb some DDT.

Based on the DDT testing and the literature search, it is evident that activated carbon has applicability in removing water soluble herbicides and pesticides of various varieties. The lethal nature of these compounds makes their control at spill sites essential. The relative removal capacity of activated carbon varies widely. Because of the low solubilities of pesticides and herbicides, carbon capacity for adsorbing them should not be a major problem, as long as adequate contact time is available.

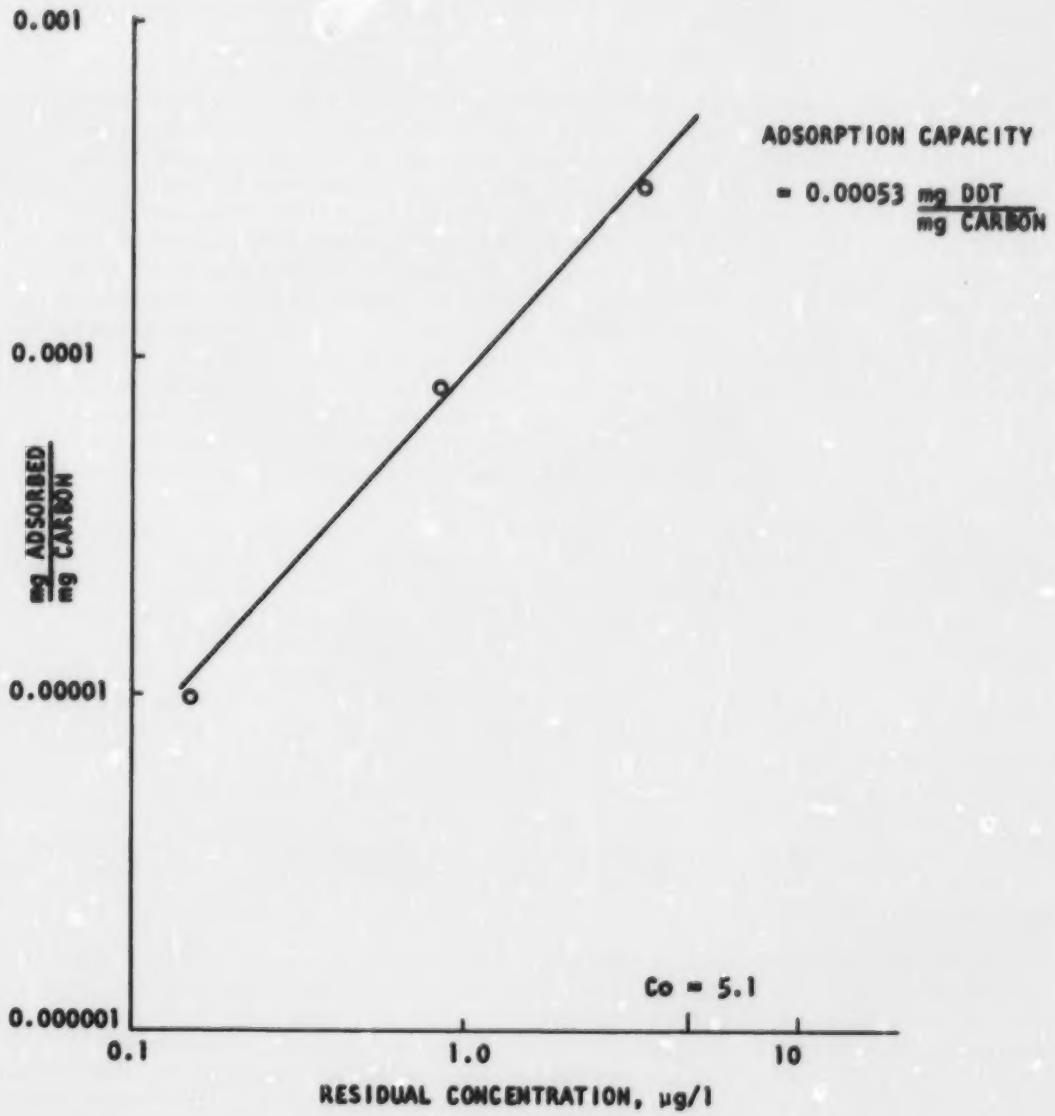


Figure 9. CARBON ISOTHERM PLOT FOR pp'-DDT

METHANOL

Dawson et al indicated that the only method of removing a methanol spill was to allow dilution and subsequent biodegradation to occur (2). Although the literature indicated poor removal of methanol by activated carbon, no concrete data could be found. Therefore, carbon tests were conducted to establish the extent of removals.

Carbon isotherm tests were performed on two solutions of methanol in tap water whose concentrations were 73 mg/l and 7253 mg/l respectively. From the results of the two isotherm tests listed in Table 7, it can easily be seen that the adsorption of methanol by the activated carbon used is very poor. A log-log plot of the data is shown in Figure 10. The adsorption capacity was estimated to be 0.028 mg/mg carbon for the 7253 mg/l methanol solution and 0.0011 mg/mg carbon for the 73 mg/l solution.

Table 7. RESULTS OF CARBON ISOTHERM TESTS PERFORMED ON METHANOL SOLUTION

Test No.	Carbon used, grams	Methanol remaining, mg/l	mg Adsorbed per mg carbon
1	0.0	7253	--
1	0.2	7220	0.0165
1	0.5	--	--
1	1.0	7120	0.0133
1	2.5	6930	0.0129
1	5.0	6810	0.0089
2	0.0	73.0	
2	0.1	74.4	
2	0.5	70.0	0.0006
2	1.0	69.5	0.00035
2	2.5	66.8	0.00025
2	5.0	59.6	0.00027

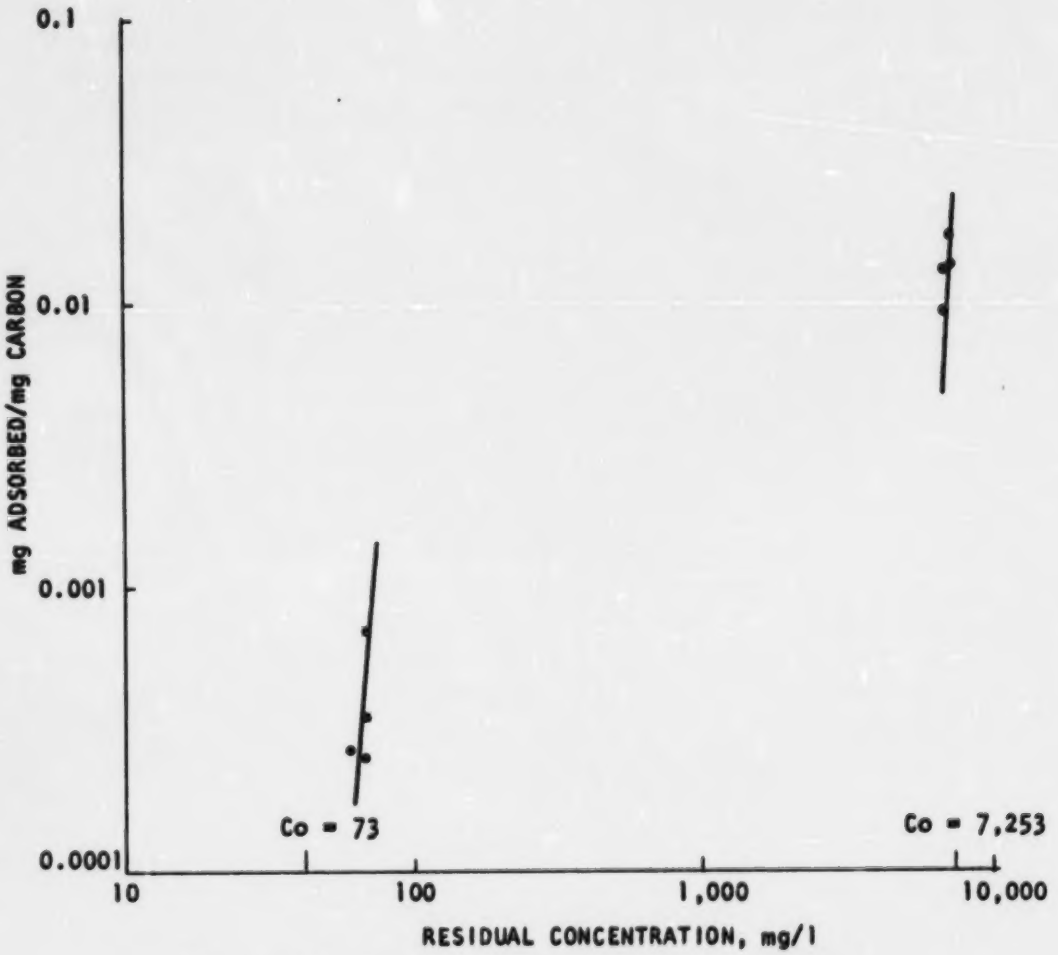


Figure 10. Carbon isotherm results for methanol

A carbon column test was also performed to verify the isotherm results. The results of this test are presented in Figure 11. The feed concentration was approximately 8000 mg/l. The adsorption capacities were found to be quite low at 0.028 and 0.926 methanol per mg carbon at hydraulic loading rates of 3.4 and 1.8 l/sec/m^2 (5 and 2.7 gpm/ft^2) respectively. Breakthrough of the carbon column was almost instantaneous. An additional carbon column test performed at a methanol concentration of 77 mg/l provided similar results which are shown in Figure 12. The adsorption capacity at this influent concentration was found to be extremely low at 0.00038 mg methanol per mg carbon at a hydraulic loading of 3.4 l/sec/m^2 (5 gpm/ft^2). Therefore, it was concluded that activated carbon is not suitable for the treatment of methanol spills.

PHENOL

Dawson et al (2) indicated that phenol spills may be successfully handled both by chemical treatment with chlorine and activated carbon adsorption. It was also indicated that if the phenol concentration was excessively high, dilution prior to treatment may be necessary. Laboratory tests on phenol were conducted both with chlorine and activated carbon.

Chlorination Tests

A test was performed to determine the approximate amount of sodium hypochlorite needed to oxidize a dilute phenol solution. The phenol solution was dosed with the desired amount of sodium hypochlorite, the pH adjusted to 8.5 and the solution allowed to react for thirty minutes. The amount of chlorine and phenol remaining was then measured. The results of these tests are listed in Table 8. It is apparent from this data that a chlorine dose of two to four times the amount of phenol present is needed for a significant reduction in phenol concentration. Chamberlin and Griffin (18) report that the amount of chlorine needed is six to ten times the phenol concentration, when treating a similar concentration of mixed phenolics. Therefore, it is concluded that chlorination of phenolic spills is unattractive not only because of the large amounts of chemical requirements but also becuase of possible chlorinated reaction products.

Table 8. RESULTS OF CHLORINATION OF PHENOL SOLUTION

	Test 1	Test 2	Test 3	Test 4
Initial phenol concentration, mg/l	117	115	114	110
Chlorine added, mg/l	109	268	495	1920
mg chlorine added/mg phenol	0.1/1	2.2/1	4.3/1	17.5/1
Final phenol concentration, mg/l	95	15	2	<0.05
Final chlorine concentration, mg/l	<1	28	0	1307
mg chlorine used/mg phenol destroyed	4.9/1	2.4/1	4.4/1	5.6/1

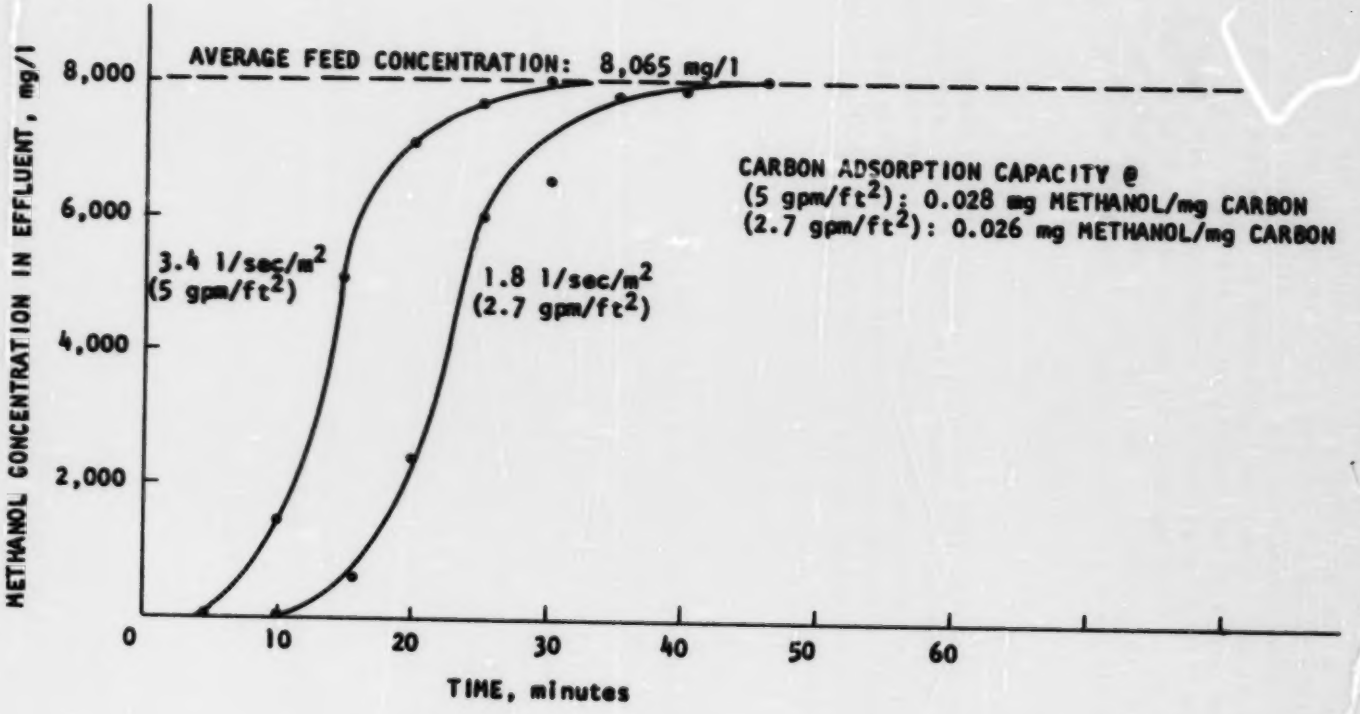


Figure 11. Carbon column tests for methanol (high concentration)

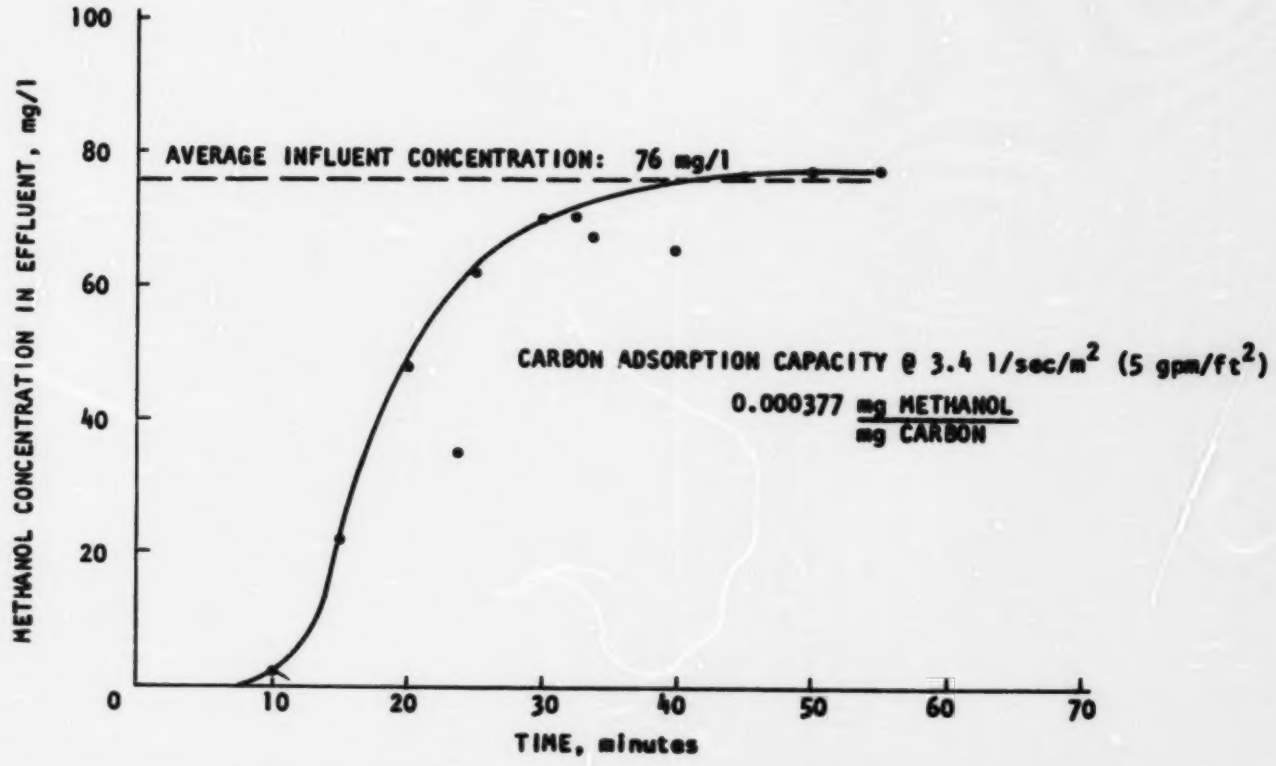


Figure 12. Carbon column test for methanol (low concentration)

Activated Carbon Adsorption

Carbon isotherm tests were performed on four solutions of phenol. The concentration of phenol was measured using the 4-amino antipyrine colorimetric method with carbon tetrachloride extraction. Three of the phenol solutions tested were solutions of phenol in Milwaukee tap water without pH adjustment; the pH of these solutions varied from 7.4 to 7.5. The pH of the fourth solution was adjusted to 10 with sodium hydroxide before the isotherm test. The results of the isotherm tests are shown in Figure 13. The results from the test performed on the high pH phenol solution were not significantly different from the results of the other tests. The adsorption capacity was estimated to be 0.41 mg/mg carbon for the 9040 mg/l phenol solution, 0.24 mg/mg carbon for 924 mg/l and the 1026 mg/l solutions and 0.14 mg/mg carbon for the 102 mg/l solution.

Activated carbon column testing was performed on a phenol solution of 9300 mg/l at a hydraulic loading rate of 3.3 l/sec/m^2 (4.9 gpm/ft^2). The results of this run are presented in Figure 14. Complete removals of phenol were achieved by carbon adsorption. The adsorption capacity was found to be Q.212 mg per mg carbon which was 47% of the carbon isotherm value of 0.45 mg/mg carbon. The above values of carbon adsorption capacities for phenol match quite closely to the data shown in Table 9 provided by the Calgon Corporation (manufacturer of Filtrasorb 400). Since sufficient data was available from the manufacturer on the carbon adsorption efficiencies at lower phenol concentrations, no further carbon column tests were conducted with phenol. From this data, it is apparent that activated carbon can effectively treat phenol spills.

Table 9. PHENOL ADSORPTION EFFICIENCIES ON FILTRASORB 400[a]

Concentration of phenol, ppm	Contact time, minutes	Phenol loading, % by weight
16	30	5 - 10
10,000	75	20 - 25
1,140	18	10 - 20
1	15	3 - 5
2,500	30	12 - 15
225	75	20
2,500	80	23

a. Data provided by Calgon Corporation.

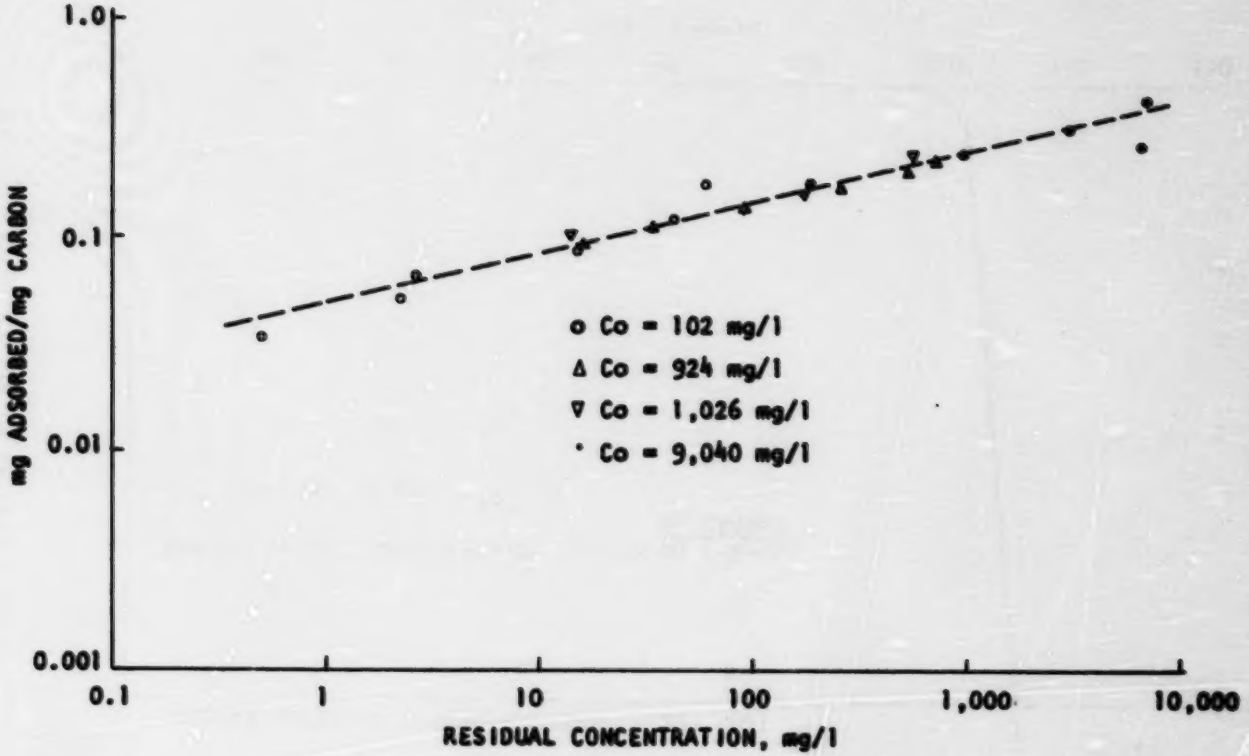


Figure 13. Carbon isotherm results for phenol

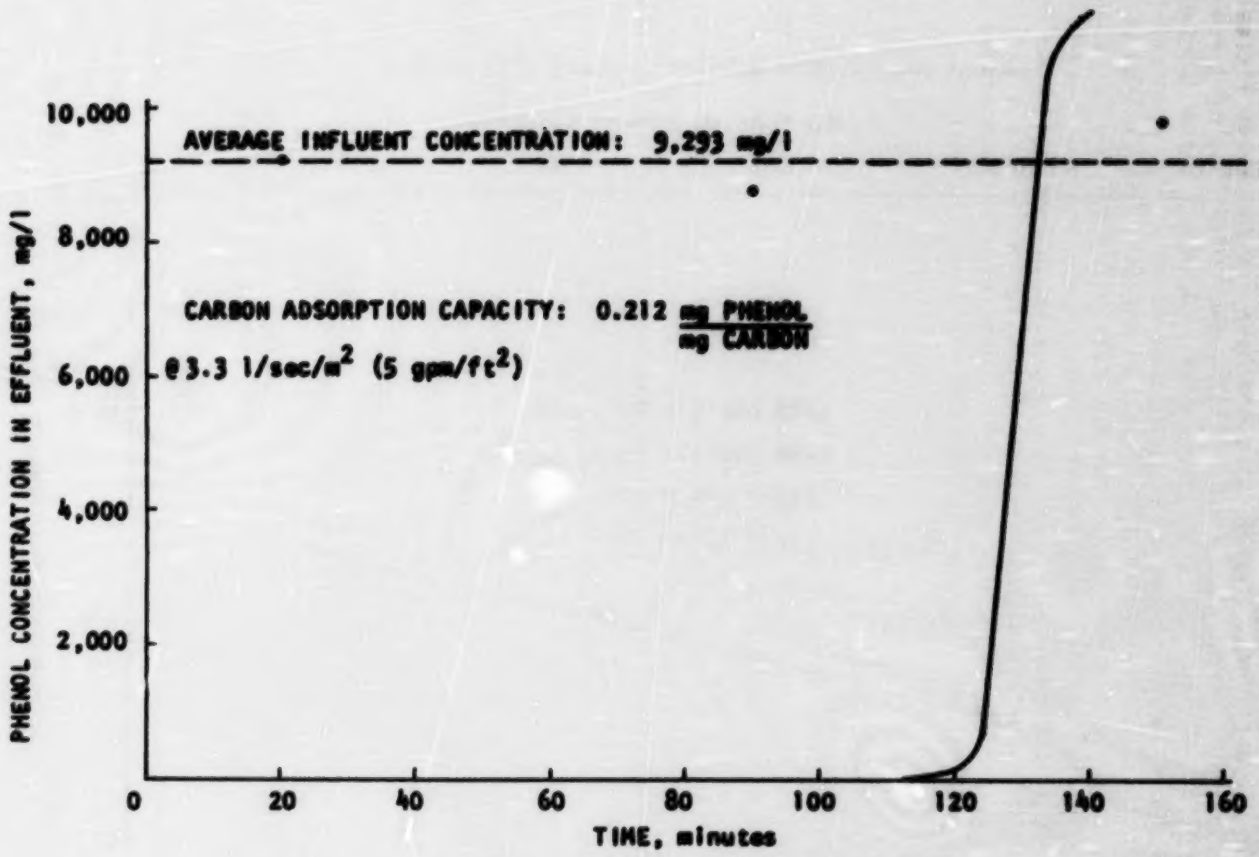


Figure 14. Carbon column test for phenol (high concentration)

ORGANO LEAD COMPOUNDS (TML AND TEL)

Both tetraethyllead (TEL) and tetramethyllead (TML) are heavy liquids having specific gravities of 100 and 165 respectively. Both these compounds are considered highly toxic and have limited solubilities in water. According to Shapiro and Frey (19), the solubility of these compounds is about 0.2 to 0.3 mg/l in water. However, higher concentrations of these compounds can occur in contaminated waters at a spill site due to emulsification or adsorption on solids. TML is also an unstable compound and generally contains 20% to 30% toluene for stability. TEL is generally difficult to recover, but can be complexed with a calcium salt or EDTA or dimercaprol (2). Laboratory tests were conducted to establish the expected concentrations of these materials at a spill site. Chemical treatment and carbon adsorption tests were then conducted on solutions containing the expected concentrations of TEL and TML.

Solubility Tests

Some preliminary tests were conducted by mixing known amounts of TML and TEL in water. In one test, approximately 5 ml of TEL was added to 750 ml tap water in a glass stoppered one liter bottle. The contents of the bottle were shaken vigorously and allowed to stand for 15 minutes to allow the TEL droplets to settle. Immediate TEL analysis of a supernatant showed a concentration of 12.8 mg/l tetraethyllead and 3.3 mg/l as ionic lead. A second sample of supernatant, removed three hours after mixing in the glass bottle, showed 6.8 mg/l tetraethyllead and 13 mg/l ionic lead. Another mixture of TEL and water was mixed several times over a period of four days and allowed to settle. The supernatant contained less than 2 mg/l of TEL but had an ionic lead content of 46.5 mg/l. Evidently the TEL was degrading during mixing and storage. Also, a highly volatile organic liquid was observed floating on the surface of the water when stored in stoppered bottles.

Shapiro and Frey (19) report that organolead compounds may be decomposed by ultraviolet light and that prolonged contact with air, especially in the presence of light, can cause gradual decompositions. A test was performed to study the effect of light and air on a TML water mixture. Approximately two liters of tap water that had been deaerated by bubbling nitrogen through it, was vigorously mixed with 2 ml TML (80% TML in toluene). The air space above the mixture was flooded with nitrogen prior to mixing and during a 20 minute settling period. After settling, portions of the supernatant were transferred to the test flasks under the following conditions:

Flask 1 - covered with foil to exclude light and stoppered to exclude air

Flask 2 - stoppered to exclude air, but exposed to light

Flask 3 - covered with foil to exclude light, but exposed to air

Flask 4 - exposed to both air and light.

The stoppered flasks were flushed with nitrogen prior to and after the addition of the samples. The stoppers were fitted with septums to allow the removal of sample by syringe for gas chromatography without exposing to air.

A fifth flask was prepared containing deaerated water and 0.5 ml TML. The flask was covered with foil and stoppered. Vigorous mixing of the TML and water was avoided to prevent the formation of suspended droplets of TML. The purpose of the fifth flask was to determine the solubility of TML in water which is in contact with excess TML.

The results of these tests are listed in Table 10. The following observations can be made:

1. The TML concentration of the solutions exposed to air dropped to less than half the concentration of solutions protected from air within two hours.

2. The TML concentration of all four test solutions decreased to about the same level after 2 days.

3. The concentration of TML in the four test solutions was approximately half the concentration of TML in the water in flask 5 after 2 days.

It appears that the decrease in TML concentration in water with time is not due entirely to exposure to light and air, or to settling of the suspended TML droplets. The dynamic nature of these solutions demonstartes that extreme care must be taken in testing them. Analysis for TML should be made as quickly as possible. In order to evaluate the maximum possible concentration levels of TML in water, about 5 ml of TML (80% TML and 20% toluene) was added to about 800 ml tap water in a glass stoppered bottle. The contents of the bottle were shaken vigorously, then allowed to stand for various periods of time to allow the large drops of TML to settle. The supernatant was then decanted for tests. The properties of the TML-water mixture were generally similar to that of the TEL-water mixtures previously tested. The major difference observed is that concentrations of TML in water were higher than the corresponding TEL concentration and ranged generally between 20-30 mg/l.

Table 10. STABILITY OF TETRAMETHYLLEAD IN WATER

	CONCENTRATION OF TML, mg/l				
	FLASK 1	FLASK 2	FLASK 3	FLASK 4	FLASK 5
Exposed to light	NO	Yes	No	Yes	No
Exposed to air	NO	No	Yes	Yes	No
Elapsed Time, Hrs.					
0.3	11.5	--	--	--	--
0.5	--	--	--	12.2	--
0.7	--	17.4	--	--	--
0.9	--	--	10.4	--	--
1.0	12.5	--	--	--	--
1.2	--	14.1	--	--	--
1.4	--	--	--	6.8	--
1.6	--	--	6.2	--	--
1.7	11.2	--	--	--	--
1.9	--	13.6	--	--	--
2.1	--	--	6.5	--	--
2.2	--	--	--	5.1	--
46.2	--	--	--	--	5.7
46.3	--	3.6	--	--	--
46.5	3.2	--	--	--	--
46.7	--	--	2.8	--	--
47.0	--	--	--	2.1	--
47.1	--	--	--	--	5.6
47.3	--	2.7	--	--	--
47.5	1.6	--	--	--	--
47.7	--	2.2	--	--	--
47.8	--	--	--	--	6.9
48.0	2.4	--	--	--	--
48.2	--	2.6	--	--	--
48.3	--	--	2.6	--	--
48.5	--	--	--	1.6	--
48.6	--	--	--	--	5.3

Treatment Tests - Tetraethyllead (TEL)

Chemical treatment tests were conducted on a sample containing 12.8 mg/l TEL. Southern bentonite clay was added to two portions of the sample to simulate dirt that might be suspended in water at a spill site. Concentrations of 500 mg/l and 1000 mg/l clay were used. Each sample was then treated with 50 mg/l ferric chloride (as $FeCl_3$), flocculated for two minutes and allowed to settle five minutes. Samples of the clarified water were taken for tetraethyllead analyses. The clarified water was then passed through a one inch deep sand filter to remove the small floc particles remaining in suspension. Samples were taken of the filtrate for tetraethyllead analyses. The results of these tests are listed in Table II. From these results, it appeared that flocculation and filtration would adequately remove tetraethyllead. However, it should be noted that the chemical treatment test results may have been influenced by the natural degradation of TEL with time, as indicated in earlier solubility tests. The increase in water soluble lead between settling and filtration tests would seem to further endorse this idea. However, the bench scale test work does indicate that TEL can be controlled by chemical clarification. The residual soluble lead may then be removed by conventional means of raising the solution pH and precipitating out the lead as hydroxide.

Table II. RESULTS OF CHEMICAL TREATMENT TESTS ON TETRAETHYLLEAD (TEL)

	Test 1	Test 2
Initial TEL concentration, mg/l	12.80	12.80
Concentration of clay, mg/l	500	1000
$FeCl_3$ dosage, mg/l	50	50
Mix time, min.	0.50	0.50
Flocculation time, min.	2	2
Settling time, min.	5	5
TEL concentration, mg/l		
After settling	0.10	<0.05
After sand filtration	<0.05	<0.05
Ionic Lead concentration, mg/l		
After settling	0.80	0.70
After sand filtration	1.00	1.50

Activated Carbon Adsorption - One attempt was made to run a carbon isotherm test on suspended TEL. The carbon isotherm test procedure was modified by preparing a dilute carbon slurry and measuring the desired amount of carbon slurry by pipet. The amounts of carbon used were 0 mg, 0.75 mg, 1.5 mg, 2.5 mg and 5 mg. A mixture of TEL and water was shaken and allowed to settle for one hour. The supernatant was used for the isotherm tests. The initial TEL concentration was 5 mg/l. After shaking one hour with 0 mg carbon, the TEL content was reduced to 0.5 mg/l. The TEL content of all the samples that were mixed with carbon was less than 0.1 mg/l.

As a result of the above described tests, it appears that only low levels of TEL may persist in waters at a spill site and that these amounts can be reduced to safe levels by chemical flocculation, settling and filtrations. The longer the length of time the TEL contaminated water is impounded, the lower the concentration of TEL is likely to be. The amount of ionic lead, however, is expected to increase. The soluble lead can be satisfactorily treated by conventional pH adjustment for the precipitation of lead hydroxide and removal of the precipitate by sedimentation and filtration.

Treatment Tests - Tetramethyllead (TML)

The results of chemical flocculation, settling and sand filtration tests on several TML-water mixtures are listed in Table 12. The TML-water mixtures used varied from 22 to 33 mg/l TML. A one hour to 2½ hour settling time was used to allow large droplets to settle after the initial mixing of the TML. Southern bentonite clay was added to the TML-water mixture in concentrations of 100,500 and 1,0000 mg/l to simulate dirt that might be suspended in the water to be treated. Each sample was then treated with 50 mg/l ferric chloride, mixed, flocculated for two minutes and allowed to settle ten minutes. Samples of the supernatant were decanted and passed through a one inch deep sand filter to remove suspended solids. Portions of the supernatant were analyzed for TML before and after sand filtration. Appreciable amounts of TML remained after chemical treatment and filtration.

Table 12. CHEMICAL FLOCCULATION, SETTLING AND FILTRATION OF TETRAMETHYLLEAD WATER

Test No.	1	2	3
Elapsed time after mixing, hrs	1	2.5	0.75
Clay added, mg/l	100	500	1,000
Ferric Chloride added, mg/l	50	50	50
Flocculation time, min	2	2	2
Settling time, min	10	101	10
TML concentration, mg/l			
initial concentration	33	22	32.5
after settling	19.2	14.1	21.3
after filtration	12.3	13.6	16.2
Toluene concentration, mg/l			
initial c-ncentration	142	114	--
after settling	106	90	101
after filtration	84	82	88

Tests were also conducted with potassium permanganate to evaluate its effect on the removal of TML. The results of these tests are shown in Table 13. The potassium permanganate dosages utilized during these tests were 50, 100 and 200 mg/l. Also, separate samples of TML-water mixture were kept exposed to light and air without the addition of $KMnO_4$ to compare the removal efficiencies with and without the addition of potassium permanganate. It was found that for all the three samples treated with 200 mg/l $KMnO_4$, the TML content of the treated water was reduced to less than 5 mg/l within 10 minutes while for the untreated samples, the TML content was between 7 and 14 mg/l after standing for one hour. The TML concentrations of the samples treated with 50 and 100 mg/l potassium permanganate decreased slowly. The effluent TML concentration for the 200 mg/l potassium permanganate treated sample did not change at all after the initial 10 minute reduction. It was evident that the potassium permanganate treatment was effective in handling TML solutions and can be utilized for the treatment of such spills.

Table 13. TREATMENT OF TETRAMETHYLLEAD WITH POTASSIUM PERMANGANATE

Test No.	1	2	3
$KMnO_4$ added, mg/l	50	100	200
TML concentrations, mg/l			
Immediately before $KMnO_4$	15.2	22.0	20.8
within 10 min after $KMnO_4$ addition	4.6	1.7	3.7
$KMnO_4$ treated sample after one hour	2.1	1.2	3.7
untreated portion of sample after one hour	7.0	9.3	14.4

Activated Carbon Adsorption - Two carbon isotherm tests were performed on a TML-water mixture. Procedure used was similar to the TEL isotherm test. The results are listed in Table 14. Substantial reductions in TML concentrations were observed in the flasks containing no carbon. This may have been due to the volatilization or degradation of the TML. In the first test, the TML concentration was 20 mg/l when the mixture was added to the isotherm test flasks. The TML concentration dropped to 1.8 mg/l after one hour of shaking in the flask containing no carbon. In the second test, the TML concentration after shaking one hour without carbon was 3.8 mg/l. A sample of the solution added to the flasks in the second test contained 12.3 mg/l TML approximately 2 hours after the isotherm test was started. Although this sample was not shaken, it was exposed to light and air during storage and it is probable that some TML was lost during this period. In each test, the concentration of TML in the flasks containing carbon was reduced to 1.5 to 2.1 mg/l. The amount of carbon used had little apparent effect on the residual TML concentration. The carbon did effect some TML reduction, though it was very small.

From the above data, it is evident that conventional coagulation and suspended solids removal treatment probably would not be effective for satisfactory removals of residual TML concentrations at a spill site. However, a combination of potassium permanganate treatment and activated carbon may provide more satisfactory treatment.

Table 14. CARBON ISOTHERM TEST RESULTS FOR TETRAMETHYLLEAD

Test No.	1[a]	1	1	1	1	2[b]	2	2	2	2	2
Carbon Concentration, mg/l	0	4.5	9.1	45.4	90.9	0	9.1	45.4	90.9	454	909
Residual Concentrations, mg/l											
TML	1.8	1.5	1.6	1.65	1.65	3.82	2.10	2.11	1.90	1.81	1.93
Toluene	42.8	43.2	40.5	30.6	25.3	72	63	--	46	10	1
Lead (as Pb)	--	--	--	--	--	46	45	45	43	41	40
Wt. Adsorbed/Wt. Carbon:											
TML ($x10^{-6}$)	--	6.7	2.2	0.3	0.2	--	18.9	3.8	2.1	0.4	0.2
Toluene ($x10^{-6}$)	--	100	79.1	37.7	24.6	--	103	--	28.5	13.8	7.8

a. Measured at start of isotherm test: TML = 20 mg/l, toluene = 47.7 mg/l
b. Measured about 2 hours after start of isotherm test: TML = 12.3; toluene = 113 mg/l

REVERSE OSMOSIS TESTS

The hazardous materials listed earlier in Table I were screened for reverse osmosis application feasibility based on available literature. Of the 20 materials listed in this table, data indicated that ten could not be treated suitably with reverse osmosis and no such treatment tests were conducted for these materials. These materials were: benzene, chlorine, chlorinated hydrocarbon pesticides, chlorosulfonic acid, organi lead compounds, phosphorus penta sulfide and styrene. The reasons for eliminat on of these materials from consideration are shown in Table 15. Generally, the reasons pertained to low solubility of the hazardous materials or expected adverse effects on the RO membranes. Among the remaining ten of the listed materials, it was indicated that sufficient application feasibility data was available from the RO membrane manufacturer for two materials and therefore, bench scale feasibility tests were conducted on only the remaining eight compounds.

Table 15. LIST OF HAZARDOUS MATERIALS ELIMINATED FOR BENCH SCALE RO TESTS BECAUSE OF THEIR PROPERTIES AND EXPECTED ADVERSE EFFECT ON MEMBRANE

Material	Reason for elimination from feasibility tests
1. Benzene	A solvent is expected to have adverse effects on the B-9 membrane. Also limited solubility in water.
2. Chlorine	Extremely low tolerance for B-9 membranes (<0.25 mg/l). Expected tolerance for cellulose acetate membranes up to <5 mg/l.
3. Chlorinated hydrocarbons	Low solubility in water.
4. Chlorosulfonic acid	Decomposes violently in contact with water.
5. Lead as in TEL & TML	Unstable compounds with low solubility in water.
6. Phosphorus pentasulfide	Decomposes with water to H_3PO_4 and H_2S. Hydrogen sulfide is expected to adversely affect the membranes.
7. Styrene	Low solubility in water.

The materials for which feasibility tests were conducted were: acetone, acetone cyanohydrine, acrylonitrile, ammonium salts, copper sulfate, formaldehyde, methanol and mercuric chloride. A summary of the results of the feasibility data is shown in Table 16. A description of the RO test procedures and terminology is presented in Appendix B. No specific change in rejection capabilities was noticed for any of the compounds tested because of a change in feed concentration. Generally, the inorganic compounds showed good rejection capabilities except mercuric chloride. The rejection of mercuric ion at 72% was found to be lower than expected since generally most divalent and trivalent ions can be rejected in

Table 16. RESULTS OF RO FEASIBILITY TESTS

	Material	Concentration, mg/l	pH	Molecular Weight	% Recovery	% Rejection By B-9 Membrane
1.	Acetone	793	7.9	58	65	69
		7,266	8.1		57	70
2.	Acetone Cyanohydrin	99	9.3	85	55	55
3.	Acrylonitrile	118	9.3	53	69	14
4.	Ammonium Nitrate					
	NH_4 as N	454	4.3 to 6.5	80	62	88
	NO_3 as N	392				89
5.	Ammonium Sulfate					
	NH_5 as N	1,920	5.6	132	55	98
	SO_4	7,060				98
6.	Copper Sulfate					
	Cu^{++}	1,440	5.0	159	88	96
		3,800	4.5		80	96
7.	Formaldehyde	8,130	7.4	30	57	20
8.	Methanol	10,000	7.5	32	64	13
9.	Mercuric Chloride					
	Hg^{++}	2,480	4 to 6	272	57	72
10.	Alum[a]					
	Al^{+++}	15	3.4	666	75	98
11.	Phenol[b]	500 to 2,000	7.0 to 9.0	94	75	55
12.	Sodium Phenylate[b]	2,500	10.7	116	75	95

a Reference 20.

b Data Supplied by Dupont Company.

NOTE: All laboratory tests conducted at 400 psi and at ambient temperature.

excess of 90%. Also during the brief test duration (24 hours) for mercuric chloride, it was noticed that the headloss across the membrane increased rapidly, suggesting high membrane fouling potential. Therefore, it was concluded that RO would not be suitable for the treatment of mercuric chloride.

Rejections for ammonium ion varied between 88% and 98% depending upon the type of anion associated with the ammonium ion. The higher rejection for ammonium ion when associated with sulfate ion was expected because of the generally better rejection characteristics of RO membranes in the presence of a divalent ion. Copper and aluminum ions were rejected in excess of 95% as expected.

The various organic compounds evaluated in this study showed considerably lower membrane rejection capabilities compared to inorganic materials. Methanol, formaldehyde and acrylonitrile showed poor rejection capabilities (13-20%) while acetone, acetone cyanohydrin and phenol showed medium separation capabilities (55 to 70%). All of the above mentioned organic compounds have a molecular weight of less than 100. Such organic compounds are generally expected to exhibit lower membrane rejection characteristics. However, it was found that the change in pH for some of these compounds can provide a marked improvement in the rejection capabilities. The pH values shown in Table 16 are considered to be the optimum values for membrane rejection. The effect of pH change was most pronounced for phenol where the conversion of phenol to sodium phenylate enhanced the rejection capability from 55% to 95% at comparable operating conditions.

From the above data it is obvious that reverse osmosis has limited applicability for the treatment of only a selected few hazardous materials such as soluble inorganics and high molecular weight organics (mol. wt. >100). Out of the 20 hazardous materials screened only eight showed medium to good removal effectiveness. Many of these spilled hazardous materials will require extensive pretreatment for the removal of particulate solids and/or other treatment such as pH adjustment, softening etc. prior to processing by RO. Furthermore, the reverse osmosis process being a high pressure process, requires relatively higher amounts of power. Both the additional pretreatment and power requirements may significantly influence the logistics support requirements in a spill situation. Therefore, it was concluded that reverse osmosis has only limited application feasibility for the treatment of hazardous materials.

SECTION VI

DESIGN CRITERIA FOR A MOBILE TREATMENT SYSTEM

Based on the laboratory evaluation of the various hazardous materials described in the preceding sections of this report, it was concluded that a flow schematic consisting of chemical reaction, flocculation, sedimentation, filtration and activated carbon adsorption would provide the most flexible mobile spills treatment system. These unit treatment processes were shown to remove the majority of the pollutants evaluated in this study. It was concluded that RO would not be a suitable treatment method for many spilled hazardous materials because of their low water solubility and adverse effect on membranes. RO could however, be utilized on a selective basis such as for the treatment of water soluble inorganics. For such selective uses, reverse osmosis equipment may be mounted on a separate mobile unit that could then be utilized in conjunction with another pretreatment unit capable of providing chemical and filtration treatments.

Figure 15 shows a schematic flow diagram of the recommended treatment system that was utilized for the design of the mobile spill response vehicle constructed under EPA Contract No. 68-01-0099. The raw waste pumping system provided on the spill response vehicle, consists of a submersible pump, an in-line booster pump and sufficient hoses to allow the deployment of the treatment system as far as 92 m (300 ft) from the spill site. The raw flow can be controlled manually by an in-line indicating flow meter and control valve. The maximum hydraulic design flow capacity of the pumping system is 12.6 l/sec (200 gpm). The vehicle is equipped with two other pumps (filter influent and backwash pumps). Any of the pumps provided on the vehicle can be used interchangeably during emergencies. The electrical power to operate the raw feed system as well as all of the other electrical requirements for the treatment system are provided by a gasoline fueled generator on the trailer. Various chemicals such as acid, lime, ferric chloride, chlorine or potassium permanganate (depending upon what substance is being treated) can be added in the reaction/flocculation tank to precipitate and/or flocculate the waste. An in-line pH indicator follows an in-line mixer and can be used for the adjustment of chemical addition for controlling pH. Provisions are also made for addition of a polymer to the suction side of the filter pump as a filtration conditioner. The waste then flows to a settling tank. The overflow from the settling tank is filtered through granular media filters followed by granular activated carbon adsorption. Sludge is removed from the sedimentation tank and stored

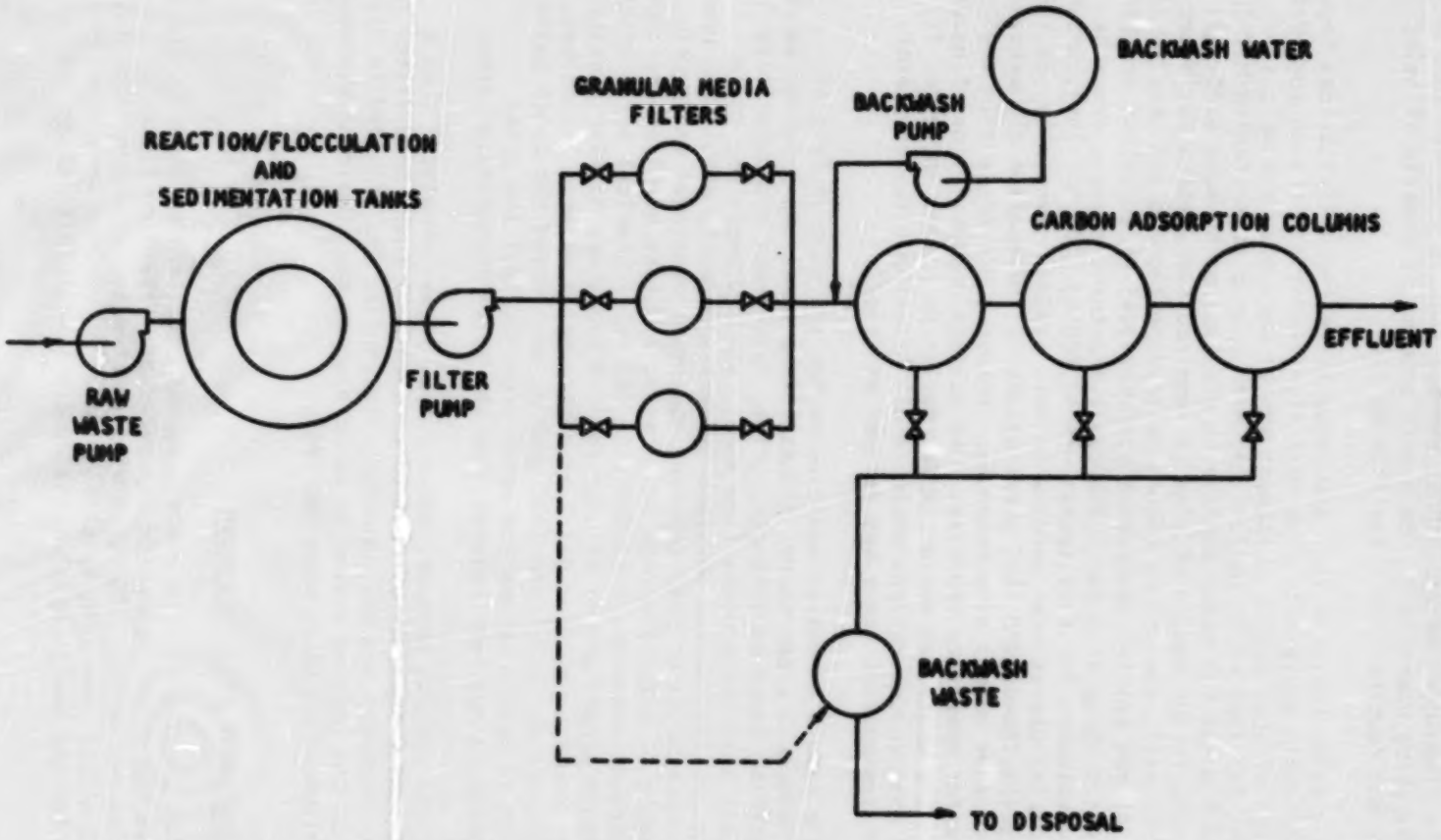


Figure 15. Waste treatment flow diagram for a mobile spill response vehicle

for ultimate disposal. An additional storage tank is provided for filter backwashing operations. Each unit process is capable of being bypassed if not required for a specific spill.

A portable treatment system must also meet the various limitations imposed upon transportable equipment. The most significant limitation involved in the design of this mobile treatment system were size and weight. A 13.7 m (45 ft) trailer in conjunction with a snub nose tractor met the required 16.8 m (55 ft) total vehicle length imposed by many states. Because a significant amount of time is required for wetting activated carbon (12-48 hrs), the carbon should be transported wet and this increases the weight of the trailer considerably, since 0.45 kg (1 lb) of dry carbon absorbs about 0.45 kg of water. The total treatment vehicle over-the-road weight selected for this system was 47,600 kg (105,000 lbs), which requires special operational permits in most states. Generally, 33,200 to 36,400 kgs (73,000-80,000 lbs) gross weight is the maximum allowable in most states under normal circumstances. However, higher weights are permissible for emergency vehicles. The maximum concentration of hazardous material in the waste was set at 1% for design purposes. However, it was recognized that concentrations would in all likelihood be much lower. The ambient temperature range was defined at 0-100°.

Based on the severe corrosion anticipated for the wide variety of hazardous materials under study, 316 stainless steel was selected as the optimum material based on strength, price, ruggedness and durability. Having established the process flow sheet, the equipment had to be designed to provide maximum possible hydraulic capacity within the space and weight constraints of over-the-road equipment mentioned previously. The importance of high flow rate in treating a dilute spill can be shown by the following example: a 19,000 l (5,000 gal.) chemical spill which empties into a stream and is diluted to 1000 parts per million creates 19 million liters (5 million gal.) of contaminated water. A treatment system operating at 12.6 l/sec (200 gpm) on an around the clock basis will require 17 days to clean the water provided that the clean effluent water is not reintroduced into the spill contaminated area.

Because of the various physical constraints, it was concluded that a maximum hydraulic capacity of 12.6 l/sec (200 gpm) could be utilized for the mobile treatment system. However, flexibility was provided in the selection of the pumping system so as to operate the treatment system at lower or higher flow rates when desirable.

Flocculation/Sedimentation System

Large rubber, portable tanks are set up next to the trailer with one tank inside the other to provide the necessary detention times for flocculation and sedimentation. A minimum chemical reaction/flocculation time of 15 minutes and sedimentation time of 60 minutes were considered necessary for the design of the treatment system. This led to the

selection of a 11,350 liter (3000 gal.) reaction/flocculation and 56,775 liters (15,000 gal.) sedimentation tank. Larger contact periods could be provided at reduced flow rates. In order to conserve valuable space on a mobile treatment system, specially built collapsible rubber tanks were selected in place of conventional rigid tanks. A flocculation/ sedimentation system was developed whereby the two 11,350 liters and 56,775 liters tanks could be utilized concentrically. This arrangement allowed a flocculation time of 15 minutes and a detention time of 60 minutes at a flow rate of 12.6 l/sec (200 gpm). The flocculation tank is placed at the center of the sedimentation tank. The raw waste is introduced at the bottom of the flocculation tank. Flocculation is achieved by hydraulic mixing via the use of two ejectors placed opposite to each other. The flocculated wastewater flows out of the center into reaction tank into the 45,400 liter (12,000 gal.) annular space of the sedimentation tank through a series of submerged orifices located around the periphery of the reaction tank. Both these tanks are cylindrical open top tanks are are supported by staves anchored into the ground. The open top tanks also permit easy accessibility for manual removal of sludge and floating materials. A photograph of the completely assembled flocculation/sedimentation tanks is shown in Figure 16.

A 11.9 l/sec (50 gpm) sludge pump and special suction fittings are provided for removal of settled and floating contaminants and a 11,350 liter (3000 gal.) rubber stave tank is provided for storage. The sludge can subsequently be pumped out of the stave tank by the sludge pump into a tank truck or other container for further treatment and/or final disposal.

Dual Media Filters

The supernatant from the sedimentation tank is drawn off by the filter pump through a submerged orifice header ring at the outside tank wall. A pneumatic level sensor in the sedimentation tank controls filter pump flow to match raw flow.

After the addition of a filtration conditioner, the sedimented effluent is pumped through three dual media filters in parallel for removal of residual suspended solids by the sand-anthracite filter media. Two in-line turbidimeters monitor the turbidity of the total filter influent and effluent from each tank. A differential pressure gauge indicates the degree of filter clogging. The filters may be taken off line individually and backwashed with air and clean system effluent stored in a 11,350 liter pillow tank. Pictures of the filter pump, filter tanks and plumbing arrangement are shown in Figures 17 and 18. The tanks are located just behind the filter and backwash pumps in the center of the trailer. The filters consist of 61 cm (24 in.) anthracite (coal, 0.9 mm effective size) over 45 cm (18 in.) fine sand (0.45 mm effective size). The

Figure 16. Completely assembled flocculation/sedimentation tank in field

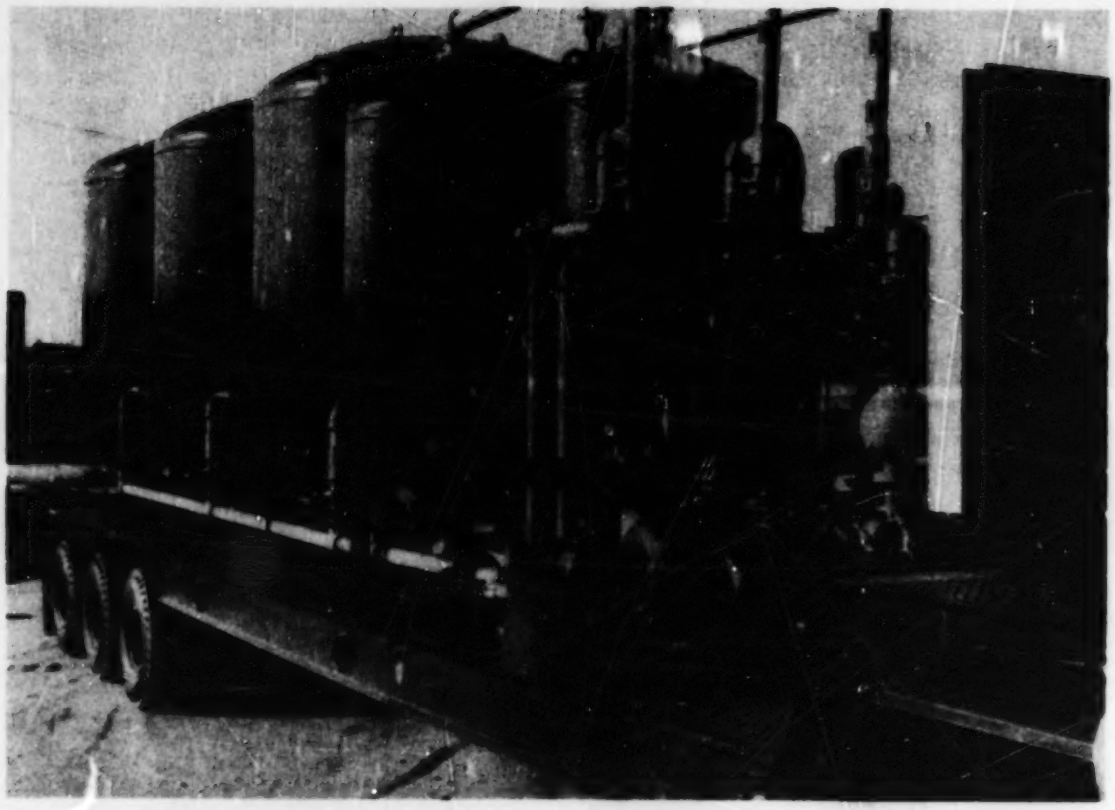


Figure 17. Photograph of the mounted dual media filters

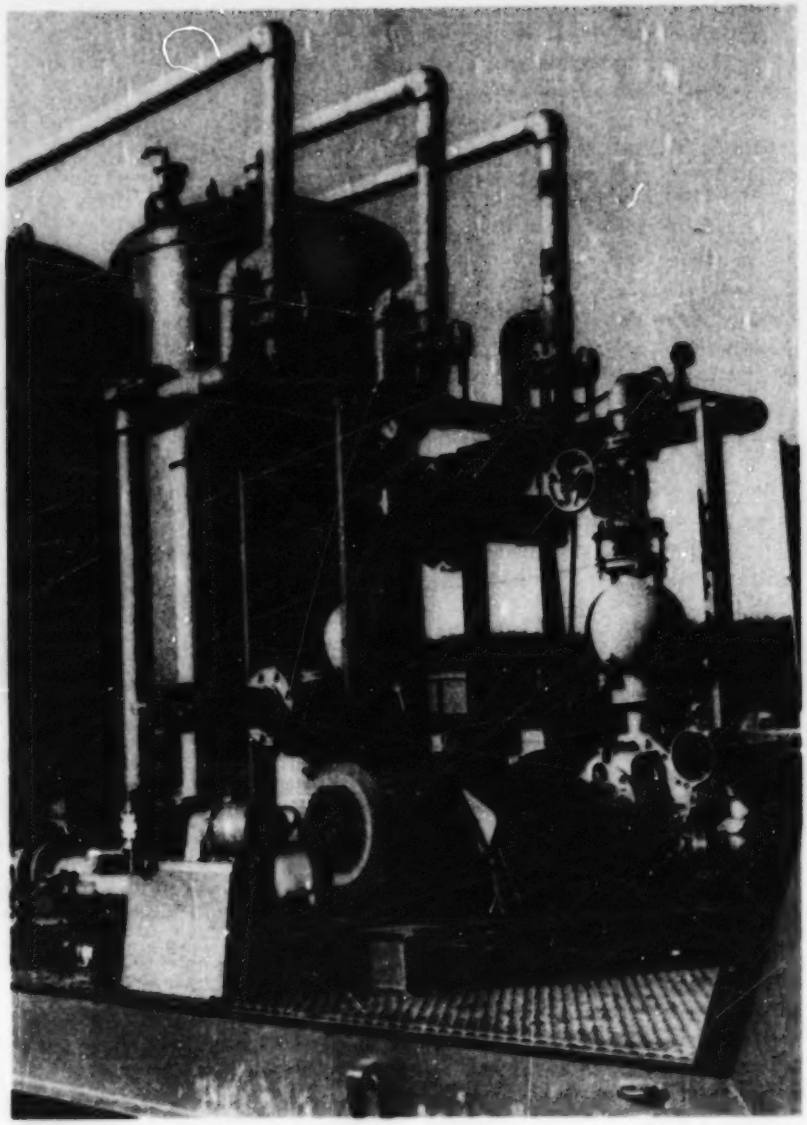


Figure 18. Closeup of the filter plumbing arrangements

filters are designed for a maximum hydraulic loading of 4.8 l/sec/m^2 (7.0 gpm/ft^2). Additional pertinent filter data for each of the three filters is given below:

Filter diameter	1.17 m (3.5 ft)
Filter area	0.893 m^3 (9.62 ft^2)
Design filtration rate	2.11 l/sec (33.5 gpm)
Maximum filtration rate	4.22 l/sec (67 gpm)
Design backwash rate	6.3 l/sec (100 gpm)
Maximum allowable differential pressure	1.06 kg/m^2 (15 psi)
Maximum tank pressure	4.93 kg cm/cm^2 (70 psi)
Depth of sand	45 cm (18 in.)
Effective size	0.5 mm
Uniformity coefficient	1.5
Quantity of sand	860 kg (1900 lbs)
Depth of coal	61 cm (24 in.)
Effective size	0.85 - 0.95
Uniformity coefficient	1.7
Quantity of coal	0.425 m^3 (15 ft^3) 0.39 ton

The dual media portion of the trailer is a completely assembled system ready for operation. A backwash system is provided to flush away the captured solids and thus the filter is continuously regenerated without the need for media replacement. The backwash waste is pumped to a 11,350 liter (3,000 gal.) stave tank. From here it can be pumped out by the sludge pump either into a tank truck for disposal or it may be reintroduced into the raw feed line and recycled through the system.

The air-backwash system is arranged so that two filters may be left on line while one is taken off line for backflushing. The flow through the system will have to be decreased during backwashing if reducing from three to two filters on-line causes a differential pressure rise. The amount of throttling-back will depend on how dirty the filters are. It is recommended to backflush before the filters get too clogged for the reason that when the clean filter is brought back on line, it will take a high percentage of the flow at an increased filtration rate. This condition could lead to unusually high effluent turbidity. One way to cause balanced filter flows with a new filter on line is to throttle the filter inlet valve somewhat to cause an "artificial" headloss across the filter which will tend to balance the flows.

Carbon Columns

The filtered effluent flows through three pressure carbon columns which may be used in parallel or in series. Altogether, they contain 19.6 m^3 (700 ft^3) of carbon. This volume represents a dry carbon weight of 8172 kg (18,000 lbs) of carbon which is the maximum possible weight that can be accommodated on the trailer because of the overall weight constraints on the mobile system. The carbon columns are designed for a hydraulic loading rate of 3.4 l/sec/m^2 (5 gpm/ft^2). Three 2.1 m (7 ft) diameter carbon columns with carbon bed depth of 1.8 m (6 ft) are provided on the mobile treatment system. The selected carbon volume of 19.6 cu m (700 cu ft) on the trailer provides a maximum contact time of 27 minutes for the three columns at a flow rate of 12.6 l/sec (200 gpm). This carbon contact time was found to be suitable for many of the hazardous materials evaluated in this study. However, when high contact times are required, these may be provided by reducing the hydraulic flow rates through the carbon columns proportionately.

The carbon tanks occupy the back one-half of the trailer as shown in the photograph of the overall system, Figure 19. The carbon columns are completely plumbed so that only valve adjustments are necessary to control the various modes of operation. The trailer is designed so that it may be transported with the carbon columns full of the wet, drained carbon (the carbon holds 0.45 kg (1 lb) for 0.45 kg of water in the drained condition). However, whether done on the site or back at the homebase, the carbon will need to be recharged either because of exhaustion, to prevent undesirable effects of mixing contaminants, or because storage with contaminants in the carbon could be hazardous. Spent carbon may be removed using the backwash pump and processed clean water stored in one of the rubber tanks for this purpose. The clear effluent water is pumped through the underdrain system of the carbon column to fluidize the bed and cause the slurry to drain out of the tank drain fitting.

When the adsorptive capacity of the carbon for the processed hazardous materials is depleted, new carbon may be installed into the tanks in the field by a slurry pumping system. Depletion time, or breakthrough, occurs simultaneously for parallel operation and sequentially for series operation. Thus all of the carbon must be replaced at once for parallel operation as opposed to one tank at a time for series operation. The tank plumbing permits a rotation of the flow sequence through the tanks for series operation. Thus, the leading tank eventually becomes the second tank in line and finally assumes the third position until breakthrough and carbon replacement, whereon it again assumes the first position. The slurry pumping system for carbon replacement utilizes a dry carbon hopper feeding an eductor through which clean effluent from the effluent storage tank is pumped to form a slurry. The slurry is pumped into the carbon column where it is dewatered by the carbon column underdrains. The water is then returned to the effluent storage tank completing the closed loop slurry pumping system.

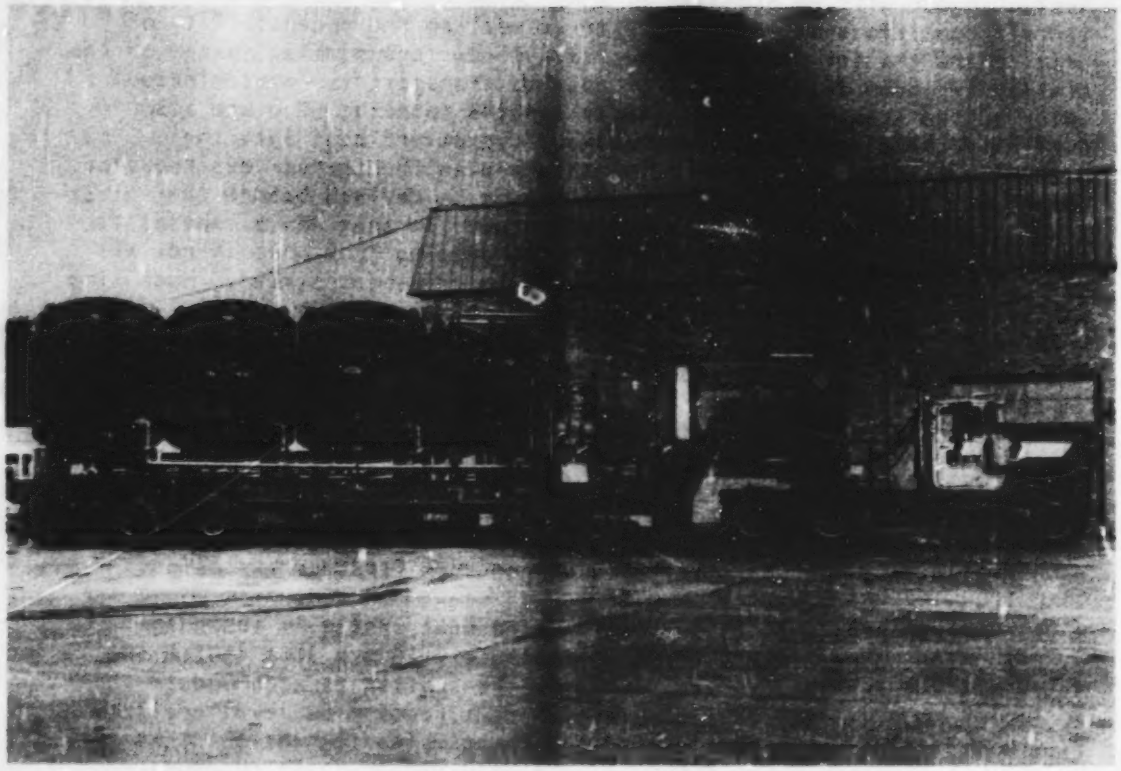


Figure 19. Carbon column as mounted on the mobile treatment system

A manual sampling valve in the carbon column inlet line and in each tank effluent line permits analysis of process water to measure removal effectiveness and to sense carbon column breakthrough.

Treatment System Layout

System layout will be dictated in many cases by the space available and terrain conditions at the spill site as well as the accessibility of driving the trailer close to the site. A raw feed pumping system on the trailer includes 92 m (300 ft) of hose to transport the contaminated water to the treatment system. Hose lengths totaling 92 m are also available for discharging the treatment system effluent into the receiving stream. It is essential that a plan for the various required locations for the raw feed submersible pump be devised before the final treatment system location is selected. In that way a central location can be selected which will allow pumping from most if not all of the planned locations without moving the treatment system. Much time and effort is lost through needless moving of the system.

The trailer location must also take into account the hazardous conditions in the immediate vicinity of the spill site. These precautions should include provisions for operator safety from contamination emitting directly from the spill site and from the treatment system itself since it functions as a reconcentrator of the spill and as such is a hazardous location. In addition, the electrical generator is powered by a gasoline fueled internal combustion engine. The separator should not be operated in areas where there is danger from fires or explosions.

A recommended plan view layout of the treatment system is shown in Figure 20. The required site is approximately 15 m x 30 m (50' x 100'). There are sufficient hose provisions on the trailer to accommodate the layout shown. It is preferable that the entire site be level and free from obstructions. In particular, the reaction/sedimentation tanks rely on being level for proper function. In this regard it is recommended that, if feasible, the selected treatment site be bulldozed so that the tank can be located on very level ground, i.e. slope less than 5 cm in 8 m (2 in. in 25 ft). Problem in supporting the large tank occurs when the slope exceeds 15 cm in 8 m (6 in. in 8 ft).

The pillow tanks should also be placed on smooth and level ground to facilitate filling and emptying. The trailer does not have to be on a level surface to function properly, but it is preferred. Front-back inclination can be corrected by the landing gear on the trailer. Side to side inclination can be corrected by skimming under the wheels and landing gear with planks. The ground level at the trailer should not be more than ten feet above the ground level at the reaction-sedimentation and effluent storage tanks because of suction limitations on the trailer-mounted, self-priming pumps which draw from these tanks.

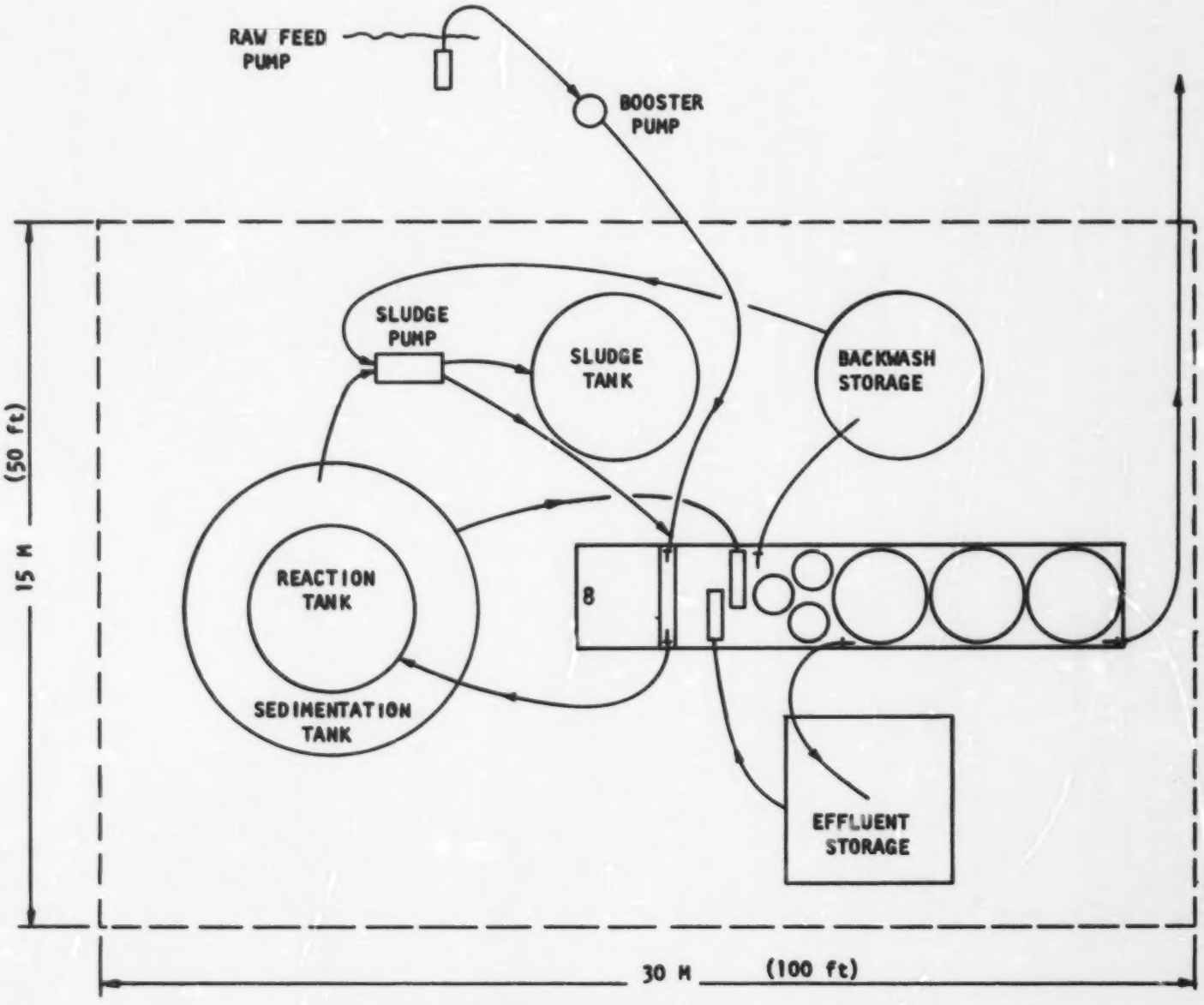


Figure 20. Recommended plan view layout of system

In assembling the system, care must be taken to allow a minimum of three feet clearance all around the trailer for access to control valves and sampling points. Ready access should be provided to the control platform which is on the right forward portion of the trailer. When laying hard-wall hoses in areas where there is vehicle traffic, either the hoses should be buried or straddled with planks which are pinned to the ground. This will prevent crushing of hoses and the resultant flow limitation.

The above described treatment system provides the necessary response vehicle that can be activated for a wide variety of spill situations in the shortest possible time. Operating experience with the vehicle and predetermined treatment procedures for specific chemicals will insure effective spill response and treatment.

Engineering Drawings, Specifications, and Operations and Maintenance Manuals

As stated in the Introduction of this report, the detailed engineering drawings and equipment procurement specifications for the fabrication of the system, as well as a set of manuals containing complete operation, repair and maintenance instructions for each component of the system, can be made available from EPA's Industrial Environmental Research Laboratory, Edison, New Jersey 08817.

SECTION VII

REFERENCES

1. Wilder, I. and LaFornara, J., "Control of Hazardous Material Spills in the Water Environment - An Overview", paper presented before the Division of Water, Air and Waste Chemistry, American Chemical Society, Washington, D.C., September 1971.

2. Dawson, G. W., Shuckrow, A. J., and Swift, W. H., "Control of Spillage of Hazardous Pollution Substances", Battelle Memorial Institute, FWQA Report 15090 FOZ 10/70 (Contract 14-12-866), November 1, 1970.

3. Fornwalt, H. J. and Hutchins, R. A., "Purifying Liquid with Activated Carbon", Chemical Engineering, April 11 and May 9, 1966.

4. Standard Methods for the Examination of Water and Wastewater, 13th Edition, American Public Health Association, Inc., New York, N.Y. 1971.

5. Methods for Chemical Analysis of Water and Wastes, Environmental Protection Agency, Analytical Quality Control Laboratory, Cincinnati, Ohio, 1971.

6. Analytical Methods for Atomic Absorption Spectrophotometry, Perkin-Elmer Corp., 1971.

7. Barnes, R. A., Atkins, P. F., Jr., and Scherger, D. A., "Ammonic Removal on a Physical-Chemical Wastewater Treatment Process", EPA Report No. EPA-RZ-72-123, prepared for Office of Research and Monitoring, USEPA, Washington, D.C., 1972.

8. King, P. H., Yeh, H. H., Warren, P. S., and Randall, C. W., "Distribution of Pesticides in Surface Waters", Journal AWWA, Vol. 61, No. 483, September 1969.

9. Lotse, E. G., Graetz, D. A., Chesters, G., Les, G. B., and Newland, L. W., "Lindane Adsorption by Lake Sediments", Environmental Science Technology, Vol. 2, No. 353, May 1968.

10. Schwart, H. G., "Adsorption of Selected Pesticides on Activated Carbon and Mineral Surfaces", Environmental Science and Technology, Vol. 2, No. 353, May 1968.

11. Huang, J. C. and Liao, C. S., "Adsorption of Pesticides by Clay Minerals", Journal Sanitary Engineering Division, Proceedings of the Chemical Society of Civil Engineers, 96:SA5, 1957 (Oct. 1970).

12. Morris, J. C. and Weber, W. J., "Adsorption of Biochemically Resistant Materials from Solution, 2", USPHS Environmental Health Series 999-WP-33, U.S. Government Printing Office, Washington, D.C., 1966.

13. Aly, O. H. and Faust, S. D., "Studies on the Removal of 2,4-D and 2,4-DCP from Surface Waters", Proceedings of the 18th Industrial Waste Conference, Purdue University, p. 6, 1963.

14. Robeck, G. G., Dostal, K. A., Cohen, J. M., and Kreissl, J. F., "Effectiveness of Water Treatment Processes in Pesticide Removal", Journal AWWA, Vol. 57, No. 181, February 1965.

15. Hartung, R. and Klinger, G. W., "Concentration of DDT by Sedimented Polluting Oils", Environmental Science and Technology, Vol. 4, No. 407, May 1970.

16. Babers, F. J., Journal, American Chemical Society, Vol. 44, 46666, 1955.

17. Bowman, M. C., Acree, F., Jr., and Corbett, M. K., Journal, Afr. Food Chem., Vol. 8, 406, 1960.

18. Chamberlain, N. S., and Griffin, A. E., "Chemical Oxidation of Phenolic Wastes with Chlorine", Sewage and Industrial Wastes", 24:750, 1962.

19. Shapiro, H. and Frey, F. W., "The Organic Compounds of Lead", Inter-Science Publisher, New York, N.Y., 1968.

20. Mason, D. G., Gupta, M. K., and Wilmoth, R. C., "Treatment of Ferrous Iron Acid Mine Drainage by Reverse Osmosis", paper presented at the Fourth Symposium on Coal Mine Drainage Research, Pittsburgh, Pennsylvania, 1972.

SECTION VIII

APPENDIX A

ANALYTICAL APPARATUS AND PROCEDURES

ANALYTICAL INSTRUMENTS AND APPARATUS

pH meter:	Beckman Model 552 Beckman Instruments Incorporated Fullerton, California
Analytical Balance:	Type H10 Mettler Instruments Corporation Heightstorm, New Jersey
Spectrophotometer:	Coleman Model 14 Coleman Instrument Company Maywood, Illinois
Atomic Absorption Spectrophotometer:	Perkin Elmer Model 403 Perkin Elmer Corp. Norwalk, Connecticut
TOC Analyzer:	Beckman 915 TOC Analyzer Beckman Instruments Co. Fullerton, California
Turbidimeter:	Hach Model 2100A Turbidimater Hach Chemical Co. Ames, Iowa
Gas Chromatograph:	Barber-Colman Series 5000 Barber-Colman Co. Rockford, Illinois

ANALYTICAL PROCEDURES

The following analyses were performed according to Standard Methods for the Examination of Water and Wastewater, (2) (Standard Methods) and Methods for the Chemical Analysis of Water and Waste (16) (WQO).

CHLORINE - P. 110 Standard Methods; Iodometric titration for total chlorine.

pH - P. 276, Method A, Standard Methods, pH meter measurement using a Beckman Model SS2 pH meter.

Sulfate - P. 286, WQO; Turbidimetric method using Hach Sulfaver.

TOC - P. 221, WQO; Beckman Model 915 TOC analyzer.

Chloride - P. 29 WQO; Mercuric nitrate titration using Hach chemicals.

Ammonia Nitrogen - P. 226 Standard Methods; Direct nesslerization using Hach colormeter.

Sulfate - P. 286 WQO; Turbidimetric method using Hach Sulfaver.

The following analyses were performed according to the methods listed in Analytical Methods for Atomic Absorption Spectrophotometry, Perkin Elmer Corp. (17), equivalent to the mentioned WQO method. The digestion procedure described on pages 86-89 of Methods for Chemical Analysis of Water and Waste was used for all metals (16). All metals were measured by atomic absorption spectroscopy.

Copper - P. 106, WQO

Lead - P. 110 WQO

Mercury - Perkin Elmer Corp.

The following analyses were performed according ot the procedures listed:

Phenol - Hach Catalog No. 10, P. 52. Chemical removal of interference followed by treatment with 4 - aminoantipyrine for color development. Color development part is based on the following procedures: Standard Methods, P. 504 and 507, or WQO, P. 232.

Nitrate Nitrogen - Hach modification of the cadmium reduction method (Standard Methods, 13th Ed., P. 458). This method measures nitrate and nitrite nitrogen. Nitrite N must also be measured and subtracted to obtain Nitrate N.

Cyanide - P. 23 Hach Catalog No. 10; A rapid method which eliminates the need for distillation and pretreatment. Cyanide is measured colorimetrically.

Nitrite Nitrogen - P. 42 Hach Catalog No. 10; Hach modification of the diazotization method using pre-packaged chemicals.

Acetone - Column - 0.6 cm x 1.8 m (¼" x 6') stainless steel column containing Poropak Q.
Column Temperature - 200°C
Carrier Gas - Helium
Detector - flame ionization detector at 235°C.
Injector - 215°C

Quantitation by peak height using standards of acetone in water.

Acetone Cyanohydrin - Column 0.6 cm x 1.8 m stainless steel column Poropak Q.
Column Temperature - 175°C and 180°C
Carrier Gas - Helium
Detector - flame ionization detector at 200°C
Injector temperature - 180°C

Quantitations using peak area determined by disc integrator

Acrylonitrile - Column - 0.6 cm x 1.8 m stainless steel column containing Poropak Q.
Column temperature - 160°C and 170°C
Carrier Gas - Helium
Detector - flame ionization detector at 170°C and 200°C
Injector - 170° - 200°C

Quantitations using peak area determined by disc integrator.

Method - Column - 0.6 cm x 1.8 m stainless steel column containing Poropak Q.
Column temperature - 140°C
Carrier Gas - Helium
Detector - flame ionization detector at 170°C
Injector - 145°C

Quantitation - using peak area determined by disc integrator.

DDT - Column - 0.6 cm x 1.8 m (¼" x 6') glass column containing 10% DC-200 on ABS.
Column temperature - 210°C.
Carrier Gas - Argon
Detector - Electron capture (Nicked-63) detector operated at 20V and 270°C.
Injector - 200°C.

Quantitation using peak area determined by disc integrator.

Extraction Procedure for DDT

Extraction of the pesticide was performed with three 15 ml portions of pesticide grade hexane (Burdick & Jackson). Extractions were performed on a 200 ml sample aliquot using a separatory funnel. The extract plus the hexane used for rinsing was dried over anhydrous sodium sulfate and reduced in volume using a warm water bath and a stream of air which had been dried and filtered through activated carbon. The hexane extract was generally reduced to a volume of about 1 ml.

The DDT in the extract was measured by gas chromatography using a Ni^{63} electron capture detector operating at 20 volts and 280°C. A ¼" x 6' coiled glass column containing 10% DC-200 on Anakrom ABS was used at 210°C. Injector temperature was 220°C. Argon was used as the carrier gas at a flow rate of about 100 cc/min. Elution time for p,p'-DDT varied from 16.5 to 19 minutes depending on the gas flow rate for the series of runs. The calibration curve was linear to about 35 µg p,p'-DDT and usable to 60 µg. Most of the samples analyzed contained less than 30 µg p,p'-DDT per injection.

TETRETHYL LEAD - Column - 0.6 cm x 1.8 m stainless steel column containing 20% Apiezon L on Chromsorb W (AW - DMCS)
Column temperature - 160°C.
Carrier gas - Helium
Detector - Hydrogen flame ionization at 185°C.
Injector - 170°C.

Samples were injected directly on the column. Quantities of tetraethyl lead were determined from peak height. Standards were prepared using pesticide grade ethyl acetate as the solvent.

TETRAMETHYLLEAD - Column - 0.6 cm x 1.8 m (¼" x 6') stainless steel column containing 20% Apiezon L on Chromosorb W (AW-DMCS)
Column temperature - 70°C and 80°C for samples.
Detector - Hydrogen flame detector at 180°C.
Injector - 150°C.

Samples were injected directly. Quantitation was by peak area using a disc integrator. Standards were prepared using pesticide grade O - Xylene as the solvent.

APPENDIX B

BENCH SCALE TEST PROCEDURES

CARBON COLUMN TEST PROCEDURE

The test equipment is shown in Figure 21.

The lead column is utilized as a surge chamber for the positive displacement piston type pump. The pump accurately meters the test solution at the desired rate. The second column is filled with Filtrasorb carbon (approximately 450 grams) prior to each test. The third column is utilized as a siphon break to prevent loss of liquid from the second column.

1. Prepare the solution to be tested.

2. Prepare carbon for testing by boiling, to remove entrapped air in the carbon pores.

3. Load the carbon as a wet slurry. Keep the column agitated with a long rod as it is being filled to prevent the formation of air pockets.

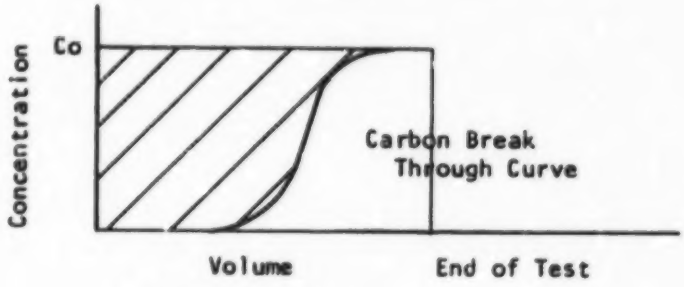
4. Start pumping test solution at predetermined rate (5-10 gpm/sq ft).

5. Take periodic column inlet and effluent sampler for analysis.

6. Plot effluent pollutant concentration -vs- time or pumped volume. This is the breakthrough curve.

7. Examine curve for time of break through, shape to determine adsorption characteristics, adsorption capacity.

8. Adsorption capacity is calculated by shaded area shown below, expressed as a fraction of the total area. This fraction times the mass of pollutant pumped over the mass of carbon utilized is the adsorption capacity.



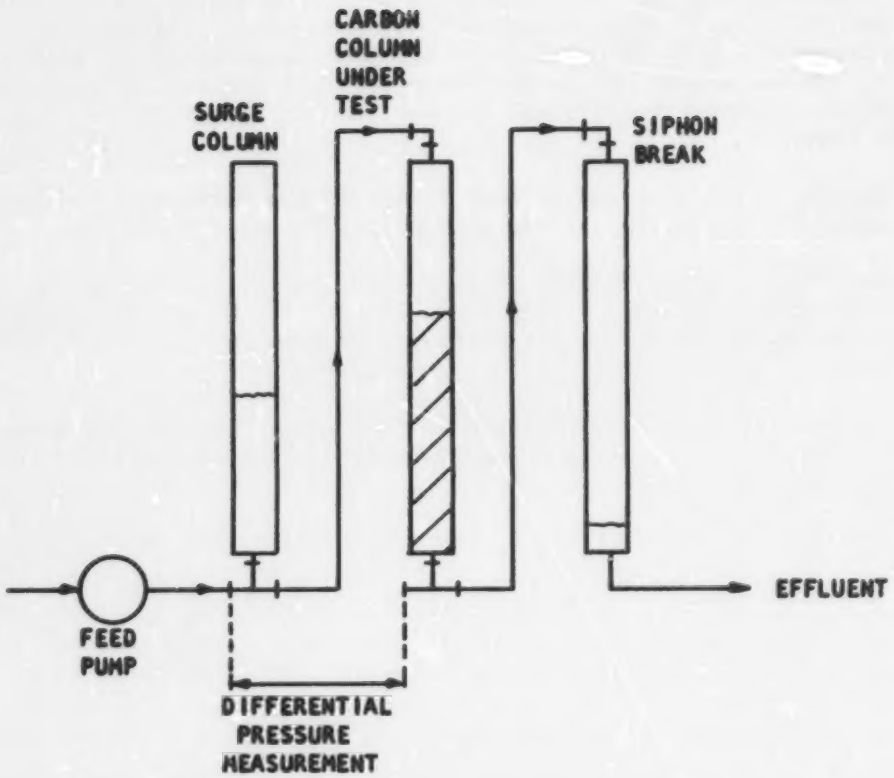


Figure 21. Laboratory carbon column test apparatus

9. Vary the depth of the carbon bed to determine the effect of different contact-time.

10. Vary the pump speed to determine the effect of hydraulic loading ($l/sec/m^2$).

PROCEDURE FOR CARBON ISOTHERM TEST

1. To each of six 250 ml erlenmeyer flasks, add one of the following weights of pulverized filtrasorb 400 activated; carbon: 0 gm, 0.2 gm, 0.5 gm, 1.0 gm, 2.5 gm, 5.0 gm. (Experience may show that other weights of carbon are preferable for the solution being tested. One of the flasks, however, should always contain no carbon.)

2. Add 100 ml of solution to each flask, stopper with a foil lined cork and vigorously agitate on a Burell Shaker for one hour.

3. Immediately after shaking is completed, remove the carbon by filtration through a 0.45 micron filter. If TOC or COD analyses are to be performed on the filtrate, be sure to wash the filters prior to use.

4. Perform the desired analysis on the filtrate. TOC or COD analyses are usually done; although analysis for a specific compound may also be done.

5. Plot log $\frac{C_o - C}{M}$ against log C_M where C_o is the weight (mg) of material in the flask containing no carbon C_M is the weight (mg) of material remaining in solution in the flask containing M mg of carbon, and M is the weight (mg) of carbon in the flask. (It is easiest to plot $\frac{C_0 - C_M}{M}$ -vs- C_M on log-log paper.) Draw a straight line through the points and extrapolate the line to C_0; the value of $\frac{C_0 - C_M}{C_M}$ at C, is an estimate of the amount (mg) of material that adsorbed by a unit weight (mg) of carbon being fed a solution containing C_o mg/100 ml material. This is known as adsorption capacity.

   For more information, see "Purifying Liquids With Activated Carbon" in April 11, 1966 Chemical Engineers by Fornwalt and Hutchins(3).

REVERSE OSMOSIS BENCH TESTS

A schematic of the RO test apparatus utilized is shown in Figure 22. It consists of a feed inventory tank with a cooling coil to adjust the temperature of the feed solution, a 10 micron cartridge filter, a high pressure pump (Hoyno), a half size B-9 membrane and the necessary valves, controls and instrumentation.

1. Make desired solution in tap water in the feed water tank.

2. Adjust cold tap water flow in the cooling water line to get temperature of feed solution between 55°F. and 65°F.

3. Turn on high pressure pump with back pressure regulator by-pass open.

4. Bring pump to desired operating pressure with pressure regulator.

5. Both the concentrate and permeate are recirculated to the feed tank and recombined.

6. Record feed water temperature, inlet pressure, outlet pressure, concentrate flowrate, feed flowrate, inlet pH, concentrate pH, product flowrate, product pH, and sample volume. (Measure flowrate by bucket and stopwatch.)

7. In order to simulate higher feed water recoveries than achievable with the system, waste the permeate until the desired recovery level is achieved. This has the effect of brine recycling, or simulating the effect of already having passed through one bank of RO membrances and then passing to the next bank. For example, wasting half of the original feed solution volume simulates having already recovered 50% of the product water.

8. Let unit run for one to four hours. Periodically record the data mentioned in Step 6.

9. Sample product water, feed water, and brine for chemical analysis.

10. Adjust the recorded flow data for temperature (68°F) and pressure (400 psi) and calculate the percent recovery salt rejection capability through the membrane in the following manner:

    % Recovery = Ratio of product flow to feed flow x 100

    % Rejection = (1-Ratio of product concentration to feed concentration) x 100.

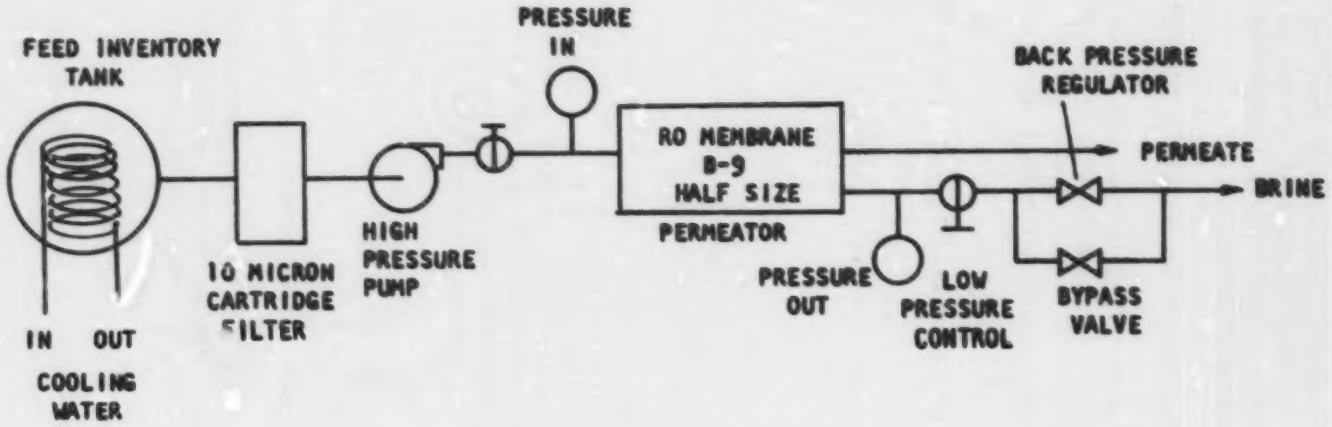


Figure 22. Schematic of reverse osmosis test apparatus